Electronic Supplementary Material (ESI) for ChemComm.

Supporting Information

Tandem Vinyl Radical Minisci-type Annulation on Pyridines: One-Pot Expeditious Access to Azaindenones

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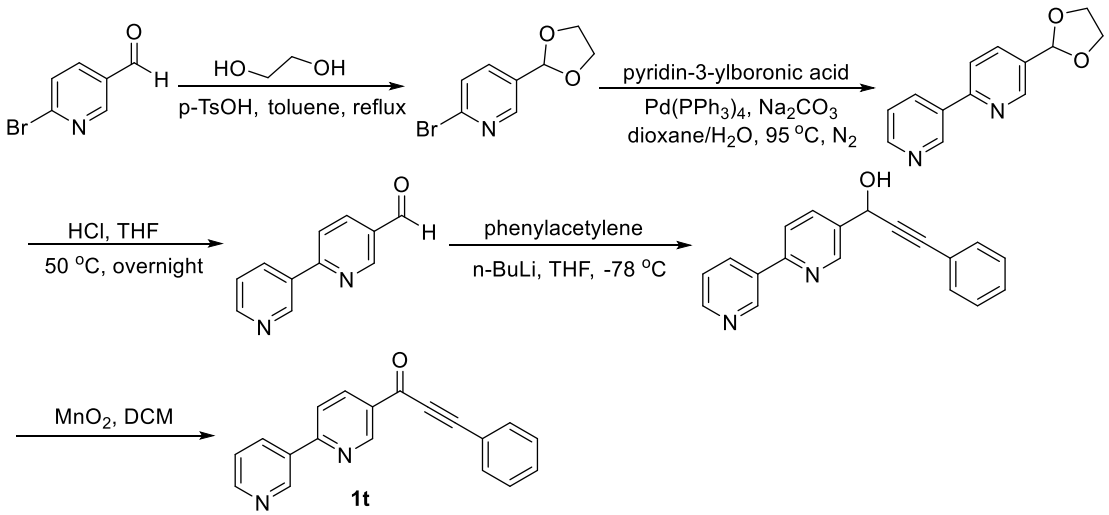
1. General experimental details

Solvents and reagents were reagent grade and used without purification unless otherwise noted. ^{1}HNMR (400 or 600 MHz) and ^{13}CNMR (100 or 150 MHz) spectra were recorded on JNM-ECE 400R or 600R spectrometers unless otherwise noted. The chemical shifts (δ) were quoted in parts per million from tetramethylsilane for ^{1}H and $CDCl_3$ for ^{13}C spectroscopy. Coupling constants are reported in Hertz (Hz). Spectral splitting patterns are designated as s, singlet; d, doublet; t, triplet; q, quartet; p, pentet; m, multiplet and br, broad. High resolution mass spectra (HRMS) were obtained with a Bruker microTOF (ESI). Single crystal was tested and recorded on a Rigaku Super Nova Single Crystal Diffractometer.

2. Preparations of Substrates

The heterocyclic ynones **1a-1s** were prepared according to the reported methods.[1,3] **1a**, **1d**, **1g**, **1h**, **1j**, **1n**, **1q-s** were known compounds and their data were in agreement with the literature data. (**1a**, **1n**, **1q**, **1r**, **1s**, see Ref. 1; **1d**, **1g**, **1h**, **1j**, see Ref. 2)

2.1 Typical procedures for the synthesis of 1t.



Add ethylene glycol (1.29 g, 20.9 mmol, 1.1 equiv.) to a suspension of 6-bromo-3-pyridine carbaldehyde (3.496 g, 19 mmol, 1.0 equiv.) and p-toluene

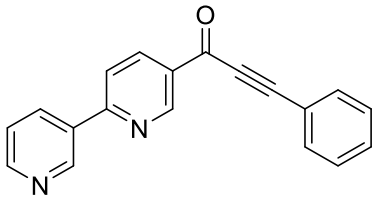
sulfonic acid (361 mg, 1.9 mmol, 0.1 equiv.) in 100 mL toluene in 250 mL flask. The mixture was reacted under Dean-Stark conditions in oil bath for 6-10 hours. After completion of the reaction (monitored by TLC), the mixture was cooled to room temperature, washed with saturated $NaHCO_3$ (30 mL) and NaCl (30 mL) aqueous solutions. The organic phase was separated, dried over anhydrous sodium sulfate and evaporated under reduced pressure. The resulting residue was purified by flash chromatography on silica gel (petroleum ether/EtOAc = 4:1) to give 2-bromo-5-(1,3-dioxolan-2-yl)pyridine as yellow oil (85%, 3.698g, 16.1 mmol).

To a 150 mL of schlenk reaction tube (with a Teflon-coated cap) equipped with a magnetic stir bar were added 2-bromo-5-(1,3-dioxolan-2-yl)pyridine (3.664 g, 16 mmol, 1.0 equiv.), pyridin-3-ylboronic acid (2.361 g, 19.2 mmol, 1.2 equiv.), Na_2CO_3 (5.088 g, 48 mmol, 3.0 equv.), $Pd(PPh_3)_4$ (924 mg, 0.8 mmol, 0.05 equiv.), H_2O (12 mL) and dioxane (68 mL). The reaction system was evacuated and purged with nitrogen for three times. The mixture was kept stirring at 80 ^oC in heated oil bath for 16 h. After completion, the mixture was cooled to room temperature, extracted with EtOAc and H_2O, and the organic phase was collected, concentrated under reduced pressure. The thus-obtained crude product was purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (v/v=4:1) as eluent to afford the bipyridyl acetal in 75% yield as white solid (2.736 g, 12 mmol).

Concentrated hydrochloric acid (12 N, 30.0 mL) was added dropwise to a solution of bipyridyl acetal (2.736 g, 12 mmol) in THF (110 mL) and water (90 mL), and the mixture was kept stirring at room temperature for 18 h. After the reaction completed, the pH of the reaction solution was adjusted to 10 with saturated potassium carbonate solution, and then extracted with EtOAc. The organic phase was dried with sodium sulfate, filtered, concentrated under reduced pressure and purified by silica gel column chromatography (PE:EA=2:1) to obtain bipyridyl aldehyde (70%, 1.545g, 8.4 mmol).

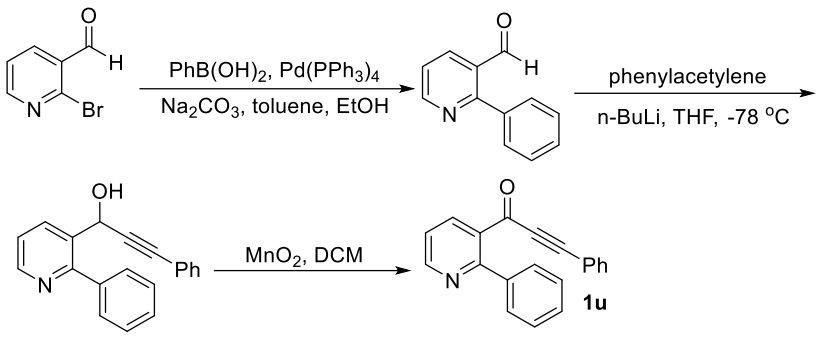
n-BuLi (2.5 M solution in n-hexane, 3.6 mL, 9 mmol, 1.07 equiv.) was added dropwise to a solution of phenylacetylene (918mg, 9 mmol, 1.07 equiv.) in anhydrous THF (20 mL) at -78 ℃ under N_2 atmosphere, stirred at this temperature for 30

minutes and then 1.0 h at room temperature. After cooling to -78 ℃, bipyridyl aldehyde (1.545g, 8.4 mmol, 1.0 equiv.) in 5 mL THF was added. The resulting solution was then warmed up to room temperature gradually and stirred for additional hours before quenched by saturated NH_4Cl aqueous solution. The mixture was extracted with ethyl ether (2 x 10 mL), and the combined organic layers were dried over anhydrous $MgSO_4$, concentrated under vacuum to afford the crude product alkynol. The crude product alkynol was dissolved in 20 mL anhydrous DCM, MnO_2 (5-10 equiv) was added, the resulting mixture was stirred at room temperature for 10 h. After the reaction completed (detected the reaction by TLC), the mixture was filtrated and the solution was concentrated under vacuum to afford the crude product, which was purified by flash chromatography on silica gel (petroleum ether/EtOAc = 1:1, V/V) to give **1t** as yellow solid (75%, 33.4% in total, 1.806g, 6.36 mmol).



1-([2,3'-bipyridin]-5-yl)-3-phenylprop-2-yn-1-one (1t). Yellow solid (1.806 g, 33.4% in total, 6.36 mmol); R_f = 0.32 (petroleum ether/ethyl acetate = 1:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.538-9.536 (m, 1H), 9.32 (s, 1H), 8.74 (s, 1H), 8.53 (dd, J_1 = 8.3 Hz, J_2 = 2.2 Hz, 1H), 8.43 (d, J = 8.0 Hz, 1H), 7.93 (d, J = 8.2 Hz, 1H), 7.72 (d, J = 7.0 Hz, 2H), 7.53 (t, J = 7.5 Hz, 1H), 7.48-7.45 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.5, 150.3, 149.8, 139.2, 133.3, 132.7, 131.3, 129.8, 129.7, 129.5, 128.9, 127.9, 126.9, 119.8, 94.8, 86.6; MS (HRMS) m/z Calcd. for $C_{19}H_{13}N_2O^+$: 285.1022, $[M+H]^+$ found : 285.1024.

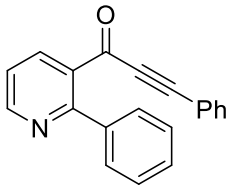
2.2 Typical procedures for the synthesis of 1u.



To a 150 mL of schlenk reaction tube (with a Teflon-coated cap) equipped with a magnetic stir bar were added toluene (100 mL), 2-bromonicotinaldehyde (1.86g, 10 mmol, 1.0 equiv.), phenylboronic acid (1.22g, 10 mmol, 1.0 equiv.) and sodium carbonate (30 mmol, 2M). Then, ethanol (6 mL) was added to this reaction mixture followed by $Pd(PPh_3)_4$ (231 mg, 0.2 mmol, 0.02 equiv.). The reaction mixture was refluxed overnight under N_2 and then diluted with water. The organic layer was separated, the aqueous layer was extracted with EtOAc (2×30 mL). The combined organic layer was washed with water (3×30 mL) and brine (1×30 mL), dried over $MgSO_4$ and filtered. The filtrate was evaporated under reduced pressure, the crude product was purified by column chromatography on silica gel (petro/EtOAc=5/1) to give the desired coupling product (60%, 1.098 g, 6 mmol).

n-BuLi (2.5 M solution in n-hexane, 2.64 mL, 6.6 mmol, 1.1 equiv.) was added dropwise to a solution of phenylacetylene (673.2 mg, 6.6 mmol, 1.1 equiv.) in anhydrous THF (15 mL) at -78 ℃ under N_2 atmosphere, stirred at -78 ℃ for 30 minutes and then 1.0 h at room temperature. After cooling to -78 ℃, the above-obtained aldehyde (1.098 g, 6.0 mmol, 1.0 equiv.) in 5 mL THF was added. The solution was then warmed up to room temperature gradually and stirred for additional hours before quenched by saturated NH_4Cl aqueous solution. The mixture was extracted with ethyl ether (2 x 10 mL), and the combined organic layers were dried over anhydrous $MgSO_4$, concentrated under vacuum to afford the crude product. The crude product was dissolved in 20 mL anhydrous DCM, MnO_2 (5-10 equiv) was added, the resulting mixture was stirred at room temperature for 10 h. After the reaction completed (detected the reaction by TLC), the mixture was filtrated and the solution was concentrated under vacuum to afford the crude product, which was

purified by flash chromatography on silica gel (petroleum ether/EtOAc = 5:1) to give **1u** as light yellow solid (70%, 42% in total, 1.188 g, 4.2 mmol).



3-phenyl-1-(2-phenylpyridin-3-yl)prop-2-yn-1-one (1u). Light yellow solid (1.188 g, 42% in total); R_f = 0.32 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.80-8.79 (m, 1H), 8.18 (dd, J_1 = 7.8 Hz, J_2 = 1.7 Hz, 1H), 7.66 (d, J = 8.0 Hz, 2H), 7.45 (t, J = 7.4 Hz, 2H), 7.39 (t, J = 7.4 Hz, 1H), 7.37-7.35 (m, 1H), 7.33 (t, J = 7.5 Hz, 1H), 7.23 (t, J = 7.8 Hz, 2H), 7.16 (d, J = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 179.7, 159.3, 151.9, 139.6, 137.8, 134.1, 133.1, 130.9, 130.1, 129.5, 128.53, 128.45, 122.0, 119.7, 94.9, 88.5; MS (HRMS) m/z Calcd. for $C_{20}H_{14}NO^+$: 284.1070, $[M+H]^+$ found : 284.1073.

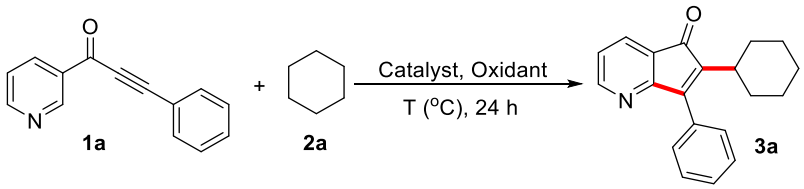
3. Optimization of Reaction Conditions

Reaction conditions were optimized using the mode reaction between 3-phenyl-1-(pyridin-3-yl)prop-2-yn-1-one (**1a**) and cyclohexane (**2a**), and the results are outlined in Table S1.

We screened Cu metal source as catalysts including CuBr, CuCl, CuI, Cu_2O, $Cu(OAc)_2$ and found product **3a** was generated in 54-70% yields (entries 2-5). Several Fe salts such as $FeCl_3$, $Fe(acac)_3$ and $FeCl_2{\cdot}4H_2O$ were also tested, and only moderate yield was obtained (entries 6-8). The yield of **3a** was only 46% in the absence of catalyst (entry 9). Some organic oxidants such as DTBP, DCP, CHP, BPO, TBPB, H_2O_2 and inorganic oxidant $K_2S_2O_8$ were screened, and TBPB was found to be the optimal oxidant in this reaction system (entries 10-16), and the reaction did not occur without an oxidant (entry 17). Further screenings indicated that 120 °C was the optimal temperature (entries 18, 19 vs 14). It was also found that 4.0 equivments of TBHP gave the best yield (entries 20, 21 vs 14) and the

optimal reaction time was 24 hours (entries 22, 23 vs 14). When cyclohexane (**2a**) was used as both reactant and solvent without the addition of other solvents the reaction proceeded most effectively (entry 24 vs 14). It should be mentioned that in contrast to the traditional protocols of Minisci reaction, the addition of an acid (TFA) did not benefit the current reaction (entry 25).

Table S1 Optimization of Reaction Conditions[a]

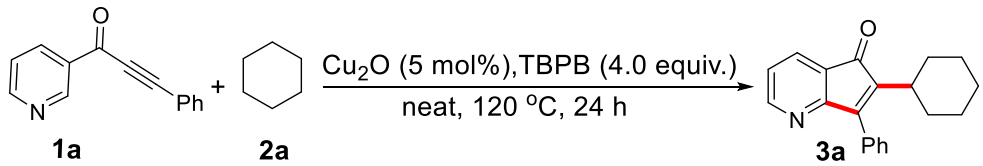


entry	catalyst	oxidant	T (°C)	yield (%)[b]
1	CuBr	TBHP[c]	120	60
2	CuCl	TBHP	120	62
3	CuI	TBHP	120	54
4	Cu_2O	TBHP	120	70
5	$Cu(OAc)_2$	TBHP	120	57
6	$FeCl_3$	TBHP	120	52
7	$Fe(acac)_3$	TBHP	120	55
8	$FeCl_2 \cdot 4H_2O$	TBHP	120	50
9	--	TBHP	120	46
10	Cu_2O	DTBP[d]	120	71
11	Cu_2O	DCP[e]	120	68
12	Cu_2O	CHP[f]	120	32
13	Cu_2O	BPO[g]	120	58
14	Cu_2O	TBPB[h]	120	78
15	Cu_2O	H_2O_2[i]	120	38
16	Cu_2O	$K_2S_2O_8$	120	0
17	Cu_2O	--	120	trace
18	Cu_2O	TBPB	110	70
19	Cu_2O	TBPB	130	77
20[j]	Cu_2O	TBPB	120	71
21[k]	Cu_2O	TBPB	120	78

22[l]	Cu_2O	TBPB	120	73
23[m]	Cu_2O	TBPB	120	79
24[n]	Cu_2O	TBPB	120	43
25[o]	Cu_2O	TBPB	120	61

[a]Reaction conditions. [b]Isolated yield. [c]TBHP = t-BuOOH (70% aq.). [d]DTBP = di-tert-butyl peroxide. [e]DCP = dicumyl peroxide. [f]CHP = cumene hydroperoxide. [g]BPO = benzoyl peroxide. [h]TBPB = tert-butyl peroxybenzoate. [i]H_2O_2 = hydrogen peroxide. [j]3.0 equivments of TBPB was used; [k]5.0 equivments of TBPB was used; [l] 18 hours; [m]30 hours; [n]THF/ Cyclohexane (V/V=1:1) was used as solvent; [o]1.2 equivments of TFA was added.

4. Typical Procedure for the Vinyl Radical Cascade Annulation (3a)



To a 10 mL of schlenk reaction tube (with a Teflon-coated cap) equipped with a magnetic stir bar were added 3-phenyl-1-(pyridin-3-yl)prop-2-yn-1-one (**1a**, 51.8 mg, 0.25 mmol, 1.0 equiv.), Cu_2O (1.2 mg, 0.0125 mmol, 0.05 equiv.) and TBPB (194.2 mg, 1.0 mmol, 4.0 equiv.). Then, 2.0 mL of cyclohexane (**2a**) was added and the mixture was kept stirring in oil bath at 120 °C for 24 h. After the reaction completed, the mixture was cooled to room temperature, extracted with saturated $NaHCO_3$ aqueous solution and EtOAc. The organic layer was then concentrated under vacuum and purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (V/V = 10:1) as eluent to afford **3a** (78%, 56.3 mg) as yellow oil.

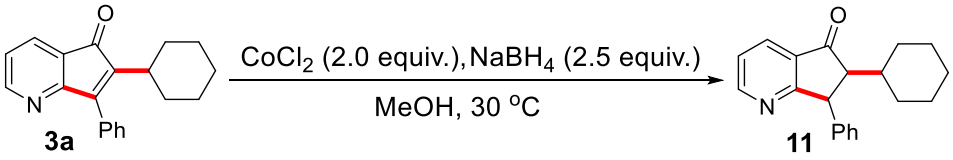
5. Procedures for Gram-Scale Synthesis of 11

a: Gram-Scale Synthesis of 3a

To a 150 mL of schlenk reaction tube (with a Teflon-coated cap) equipped with a magnetic stir bar was added 3-phenyl-1-(pyridin-3-yl)prop-2-yn-1-one (**1a**, 5 mmol, 1.036 g), Cu_2O (0.25 mmol, 24 mg), TBPB (20 mmol, 3.884 g), Then, 30 mL of cyclohexane (**2a**) was added and the mixture was stirred in oil bath at 120 °C for 24 h.

After completion, the mixture was cooled to room temperature, extracted with saturated $NaHCO_3$ aqueous and EtOAc, concentrated under reduced pressure, then purified by flash column chromatography on silica gel with petroleum [eluent: 10:1 (v/v) of petroleum ether/ethyl acetate] to afford the desired product **3a** as yellow oil in 63% yield (910.4 mg).

b: Gram-Scale Synthesis of 11

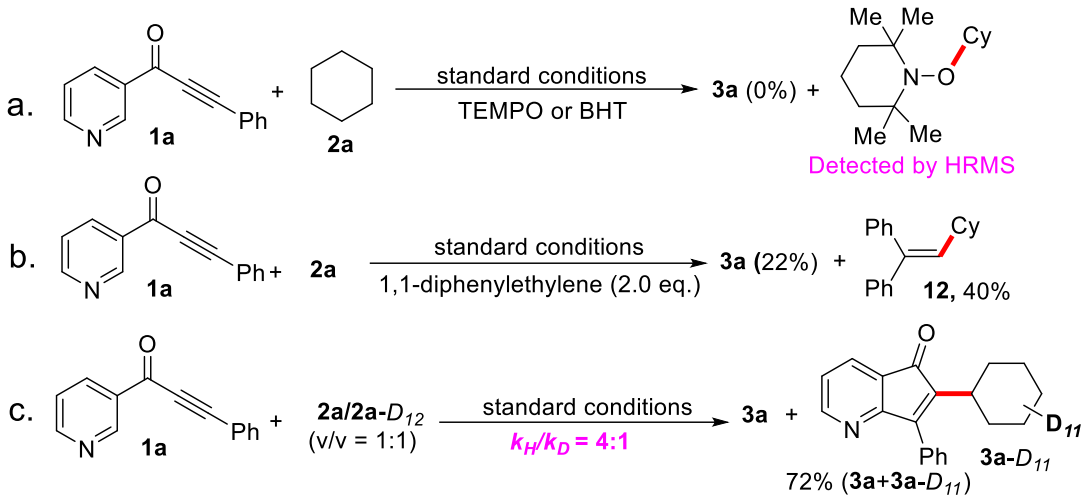


The reduction was carried out in CH_3OH with a ratio of **3a**/$CoCl_2$/$NaBH_4$ of 1:2:2.5 according to the literature[4]. To a 500-mL round bottom flasks with a magnetic stir bar was added **3a** (910.4 mg, 3.15 mmol, 1.0 equiv.) and 300 mL CH_3OH, the mixture stirred for 10 minutes. $CoCl_2 \cdot 6H_2O$ (1.498 g, 6.3 mmol, 2.0 equiv.) was added, the solutions turned deep pink and continued stirring for 30 minutes. Then, $NaBH_4$ (297.9 mg, 7.875 mmol, 2.5 equiv.) was added to the reaction mixture in portions. After completed (monitored by TLC), HCl (6N) was added, and the mixture was stirred for 1 h. Saturated Na_2CO_3 aqueous solution was added until pH = 10. After that, the mixture was extracted with EtOAc (100 mL x3). The organic layers were combined and washed with water (100 mL), concentrated under vacuum and then purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (V/V = 4:1) as eluent to afford **11** (843.3 mg, 92%, 58% in total) as colorless oil.

6. Mechanistic investigation

The mechanistic investigation included trapping of cyclohexyl radical and isotope labeling experiments, and the results was outlined in Scheme S1.

6.1 Results of control experiments



Scheme S1 control experiments

6.2 Trapping of cyclohexyl radical

6.2.1 Trapping by TEMPO

The reaction was performed according to the general procedure except for the addition of 2.5 equivalents of radical scavenger (2,2,6,6-tetramethylpiperidinoxy, TEMPO). After the reaction completed, the reaction mixture was detected by ESI-HRMS. The adduct of cyclohexyl radical with TEMPO was detected, as shown in Figure S1.

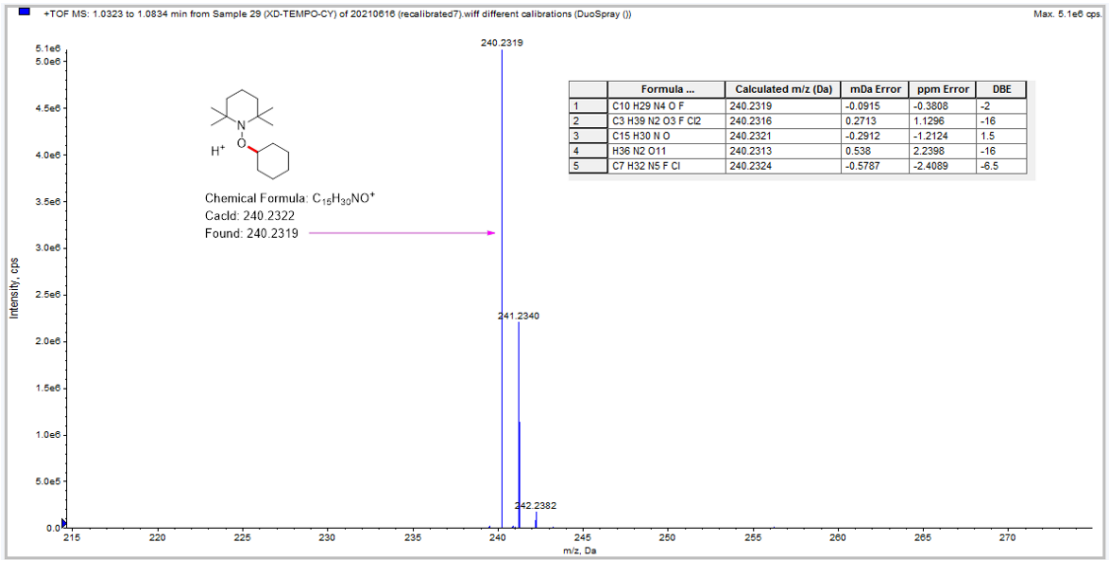
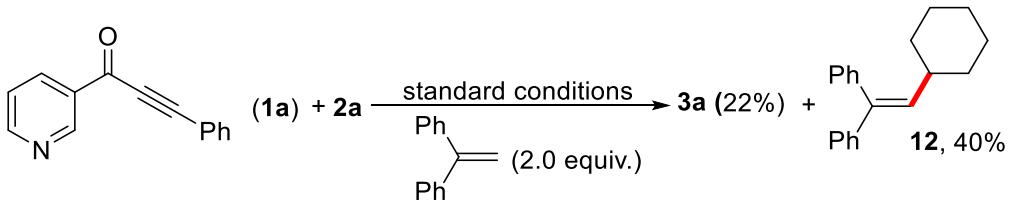


Figure S1. HRMS spectra of TEMPO-cyclohexyl adduct

6.2.2 Trapping by 1,1-diphenylethylene



The reaction was performed according to the general procedure except for the additional addition of 1,1-diphenylethylene (91 mg, 0.5 mmol, 2.0 equiv.). The mixture was stirred in oil bath at 120 ^{o}C for 24 h. After that, the mixture was cooled to room temperature, and purified by column chromatography (eluent: petroleum ether/ethyl acetate = 20/1, v/v) to afford **3a** in yield of 22% and the adduct of cyclohexyl radical with 1,1-diphenylethylene in 40% yield. The NMR spectra of **12** were outlined in Figure S2.

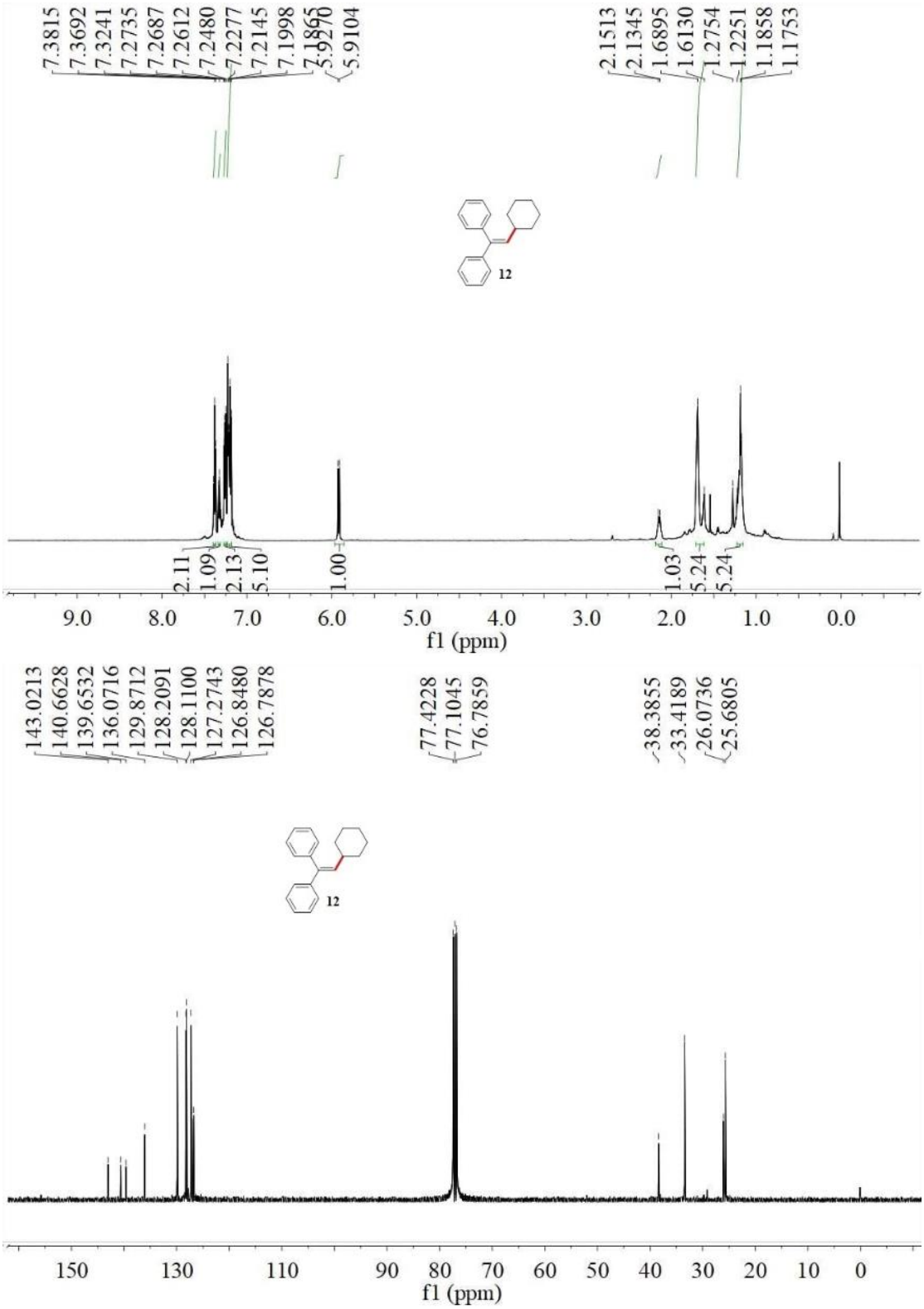
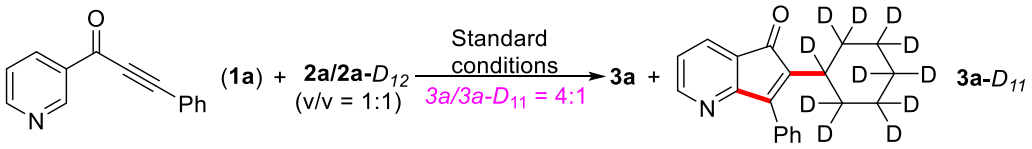


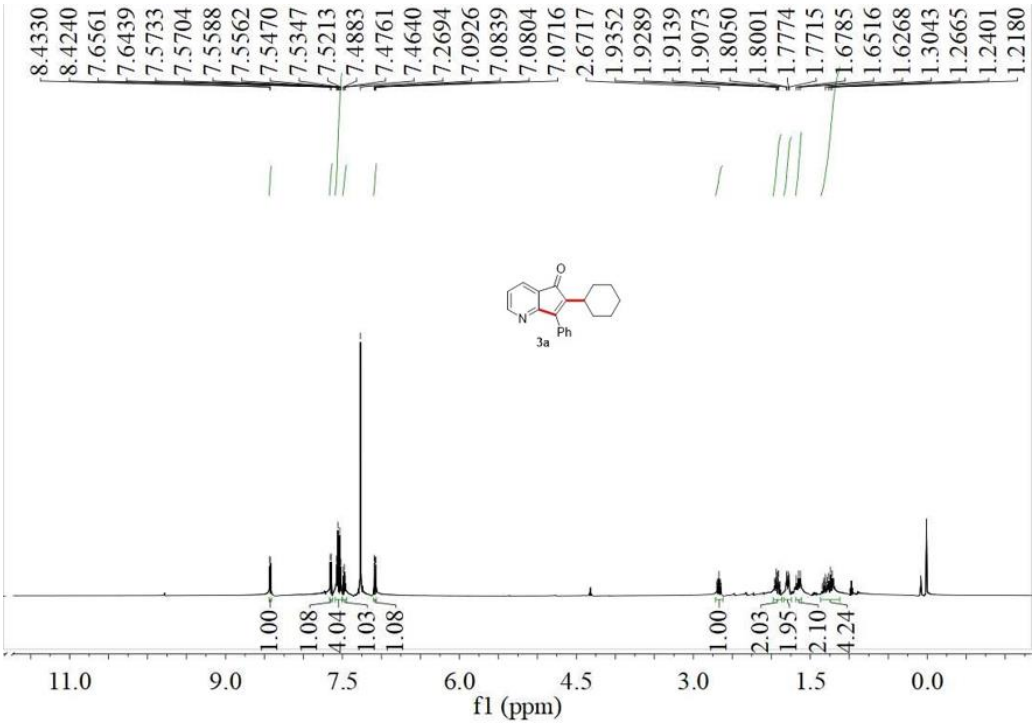
Figure S2 ^{1}H NMR and ^{13}C NMR spectrum of 12

6.3 Isotope labeling experiments



(**3a**+**3a**-D_{11}) Yield = 72%

The reaction was performed according to the general procedure except that the mixture of cyclohexane (1.0 mL) and D_{12}- cyclohexane (1.0 mL) was used instead of cyclohexane (2.0 mL). The mixture was kept stirring in oil bath at 120 ^{o}C for 24 h. After completion, the mixture was cooled to room temperature and purified by flash column chromatography on silica gel with petroleum ether/ethyl acetate (v/v = 10:1) as eluent to afford the product (**3a**+D_{11}-**3a**) in 72%. The ratio of hydrogen to deuterium (H/D) was determined from the ^{1}H NMR (as outlined in Figure S3).



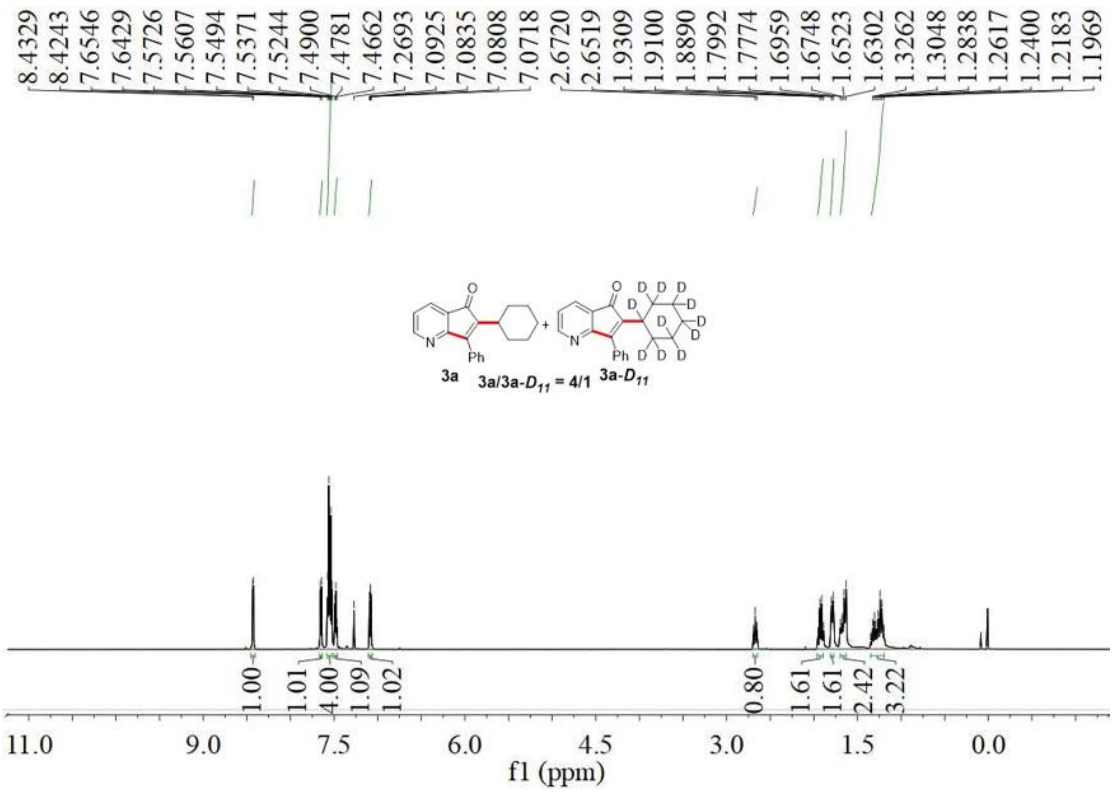


Figure S3. ^{1}H NMR spectrum of 3a, and a mixture of (3a+D_{11}-3a)

7. X-ray single crystal structure and data of 3t (CCDC 2112049)

CCDC 2112049 contains the supplementary crystallographic data (**3t**) for this paper. These data can be obtained free of charge via https://www.ccdc.cam.ac.uk/structures/Search?Ccdcid=2112049&DatabaseToSearch=Published or by emailing data_request@ccdc.cam.ac.uk

Solvent system and method for crystal growth: the cultivation of single crystal (**3t**) was in nuclear magnet tube with the slowly volatilization of deuterated chloroform.

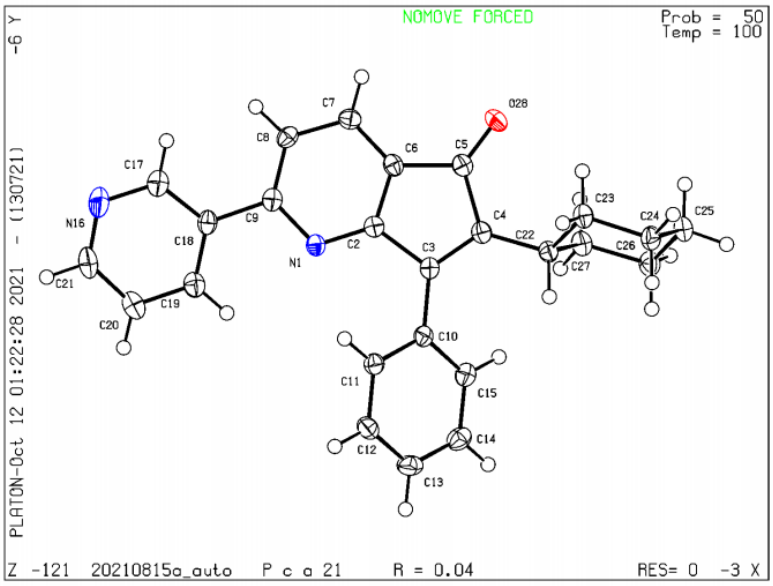


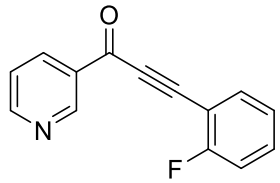
Figure S4. Ellipsoid form showing in the single crystal structure of 3t.

Displacement ellipsoids are scaled to the 50% probability level.

	3x
CCDC NO.	2112049
Description	block
color	fluorescent light colourless
from solution	$CDCl_3$
empirical formula	$C_{25}H_{22}N_2O$
Mr	366.44
crystal size (mm^3)	0.12×0.1×0.2
crystal system	orthorhombic
space group	P ca21
a [Å]	22.1929 (4)
b [Å]	12.0792 (3)
c [Å]	7.0699 (2)
α [deg]	90

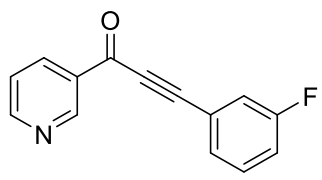
β [deg]	90
γ[deg]	90
V/ [Å3]	1895.25(8)
d/[g/cm3]	1.284
Z	4
T [K]	100
R1, wR2 I > 2ó(I)	0.0377；0.0949
R1, wR (all data)	0.0363；0.0935
quality of fit	1.051

8. Characterization data for the new compounds (substrates and products)



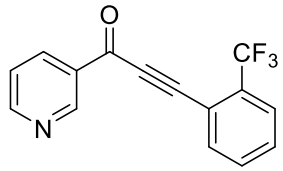
3-(2-fluorophenyl)-1-(pyridin-3-yl)prop-2-yn-1-one (1b)

Yellow solid (731.3 mg, 65%); R_f = 0.33 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.46 (s, 1H), 8.85 (s, 1H), 8.46 (d, *J* = 8.0 Hz, 1H), 7.69-766 (m, 1H), 7.53-7.47 (m, 2H), 7.24-7.18 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.3, 164.0 (d, *J* = 254.5 Hz), 154.4, 151.5, 136.5, 135.0, 133.4 (d, *J* = 8.5 Hz), 124.6 (d, *J* = 3.9 Hz), 123.7, 116.1 (d, *J* = 20.3 Hz), 108.7, 108.6, 90.8, 87.9; MS (HRMS) m/z Calcd. for $C_{14}H_9FNO^+$: 226.0663, $[M+H]^+$ found : 226.0659.



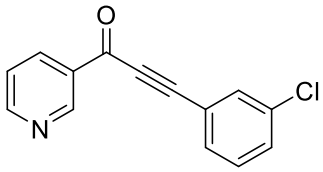
3-(3-fluorophenyl)-1-(pyridin-3-yl)prop-2-yn-1-one (1c)

White solid (765.0 mg, 68%); R_f = 0.34 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.43 (s, 1H), 8.862-8.855 (m, 1H), 8.43-8.41 (m, 1H), 7.50-7.48 (m, 2H), 7.44-7.41 (m, 1H), 7.38 (d, *J* = 8.7 Hz, 1H), 7.25-7.21 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.3, 162.4 (d, *J* = 247.2 Hz), 154.5, 151.5, 136.3, 132.1, 130.6 (d, *J* = 8.6 Hz), 129.1 (d, *J* = 3.5 Hz), 123.7, 121.4 (d, *J* = 9.3 Hz), 119.9 (d, *J* = 23.3 Hz), 118.8 (d, *J* = 21.1 Hz), 92.7, 86.5; MS (HRMS) m/z Calcd. for $C_{14}H_9FNO^+$: 226.0663, $[M+H]^+$ found : 226.0668.



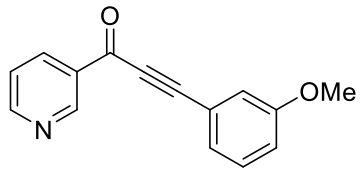
1-(pyridin-3-yl)-3-(2-(trifluoromethyl)phenyl)prop-2-yn-1-one (1e)

Yellow solid (797.5 mg, 58%); R_f = 0.33 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.42 (s, 1H), 8.86-8.85 (m, 1H), 8.45 (d, *J* = 7.9 Hz, 1H), 7.87 (d, *J* = 6.8 Hz, 1H), 7.78 (d, *J* = 8.5 Hz, 1H), 7.65-7.61 (m, 2H), 7.50-7.48 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 176.3, 154.5, 151.3, 136.5, 136.0, 132.7 (q, *J* = 31.4 Hz), 132.2, 132.0, 131.0, 126.4 (q, *J* = 5.0 Hz), 123.7, 123.3 (q, *J* = 272.0 Hz), 117.9 (q, *J* = 2.1 Hz), 90.4, 88.9; MS (HRMS) m/z Calcd. for $C_{15}H_9F_3NO^+$: 276.0631, $[M+H]^+$ found : 276.0638.



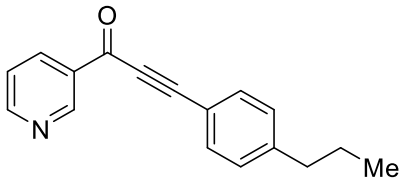
3-(3-chlorophenyl)-1-(pyridin-3-yl)prop-2-yn-1-one (1f)

Yellow solid (759.2 mg, 63%); R_f = 0.33 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.43 (s, 1H), 8.87-8.86 (m, 1H), 8.42 (d, *J* = 7.9 Hz, 1H), 7.68 (s, 1H), 7.59 (d, *J* = 7.7 Hz, 1H), 7.50-7.48 (m, 2H), 7.39 (t, *J* = 7.9 Hz, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.2, 154.5, 151.5, 136.3, 134.9, 132.9, 132.1, 131.6, 131.3, 130.2, 123.7, 121.4, 92.5, 86.8; MS (HRMS) m/z Calcd. for $C_{14}H_9ClNO^+$: 242.0367, $[M+H]^+$ found : 242.0370.



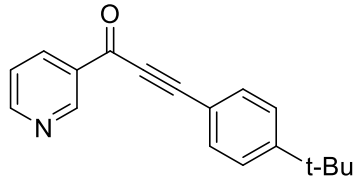
3-(3-methoxyphenyl)-1-(pyridin-3-yl)prop-2-yn-1-one (1i)

Yellow solid (829.5 mg, 70%); R_f = 0.34 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 9.45 (s, 1H), 8.852-8.845 (m, 1H), 8.43 (d, J = 7.9 Hz, 1H), 7.49-7.47 (m, 1H), 7.35 (t, J = 7.9 Hz, 1H), 7.30 (t, J = 7.5 Hz, 1H), 7.20 (s, 1H), 7.07 (dd, J_1 = 8.1 Hz, J_2 = 2.1 Hz, 1H), 3.85 (s, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 176.5, 159.6, 154.4, 151.6, 136.3, 132.3, 130.0, 125.9, 123.7, 120.6, 118.2, 117.7, 94.7, 86.0, 55.6; MS (HRMS) m/z Calcd. for $C_{15}H_{12}NO_2^+$: 238.0863, $[M+H]^+$ found : 238.0857.



3-(4-propylphenyl)-1-(pyridin-3-yl)prop-2-yn-1-one (1k)

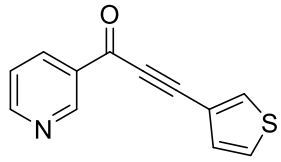
Yellow oli (647.5 mg, 52%); R_f = 0.34 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.41 (s, 1H), 8.81-8.80 (m, 1H), 8.40 (td, J_1 = 7.9 Hz, J_2 = 2.0 Hz, 1H), 7.58 (d, J = 8.2 Hz, 2H), 7.45-7.43 (m, 1H), 7.21 (d, J = 8.1 Hz, 2H), 2.60 (t, J = 7.6 Hz, 2H), 1.66-1.60 (m, 2H), 0.92 (t, J = 7.4 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.5, 154.2, 151.4, 146.9, 136.3, 133.4, 132.4, 129.1, 123.6, 116.7, 95.6, 86.3, 38.2, 24.2, 13.8; MS (HRMS) m/z Calcd. for $C_{17}H_{16}NO^+$: 250.1226, $[M+H]^+$ found : 250.1229.



3-(4-(tert-butyl)phenyl)-1-(pyridin-3-yl)prop-2-yn-1-one (1l)

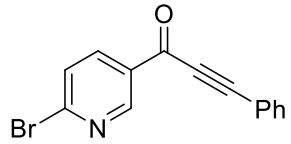
Yellow solid (723.3 mg, 55%); R_f = 0.34 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.45 (s, 1H), 8.84-8.83 (m, 1H), 8.43 (d, J = 7.9 Hz, 1H), 7.64 (d, J = 8.3 Hz, 2H), 7.49-7.45 (m, 3H), 1.34 (s, 9H); ^{13}C NMR ($CDCl_3$,

150 MHz) δ (ppm): 176.4, 155.3, 154.0, 151.4, 136.4, 133.3, 132.5, 126.0, 123.7, 116.5, 95.7, 86.3, 35.3, 31.1; MS (HRMS) m/z Calcd. for $C_{18}H_{18}NO^+$: 264.1383, $[M+H]^+$ found : 264.1387.



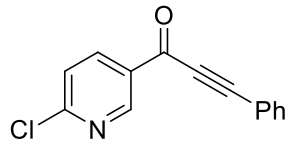
1-(pyridin-3-yl)-3-(thiophen-3-yl)prop-2-yn-1-one (1m)

Yellow solid (511.2 mg, 48%); R_f = 0.33 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.42 (s, 1H), 8.842-8.835 (m, 1H), 8.41 (d, *J* = 7.9 Hz, 1H), 7.90 (s, 1H), 7.48-7.46 (m, 1H), 7.41-7.39 (m, 1H), 7.34 (d, *J* = 5.1 Hz, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.5, 154.3, 151.5, 136.2, 134.8, 132.3, 130.4, 126.6, 123.6, 119.0, 90.2, 86.8; MS (HRMS) m/z Calcd. for $C_{12}H_8NOS^+$: 214.0321, $[M+H]^+$ found : 214.0315.



1-(6-bromopyridin-3-yl)-3-phenylprop-2-yn-1-one (1o)

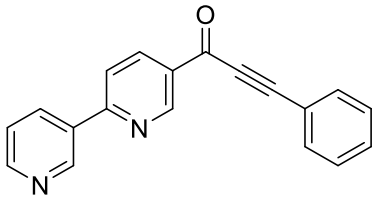
Yellow solid (954.8 mg, 67%); R_f = 0.32 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.15 (s, 1H), 8.24 (d, *J* = 8.3 Hz, 1H), 7.66 (d, *J* = 7.8 Hz, 2H), 7.64 (d, *J* = 8.3 Hz, 1H), 7.51 (t, *J* = 7.5 Hz, 1H), 7.43 (t, *J* = 7.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 175.3, 151.9, 147.9, 138.2, 133.4, 131.5, 128.9, 128.4, 119.4, 95.3, 86.2; MS (HRMS) m/z Calcd. for $C_{14}H_9BrNO^+$: 285.9862, $[M+H]^+$ found : 285.9865.



1-(6-chloropyridin-3-yl)-3-phenylprop-2-yn-1-one (1p)

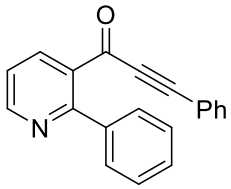
Yellow solid (735.1 mg, 61%); R_f = 0.32 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.23 (s, 1H), 8.38 (d, *J* = 8.2 Hz, 1H), 7.70 (d, *J* = 7.6 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 1H), 7.50 (d, *J* = 8.3 Hz, 1H), 7.46 (t, *J* = 7.7 Hz, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 175.1, 156.6, 151.8, 138.7, 133.4, 131.6, 131.3,

129.0, 124.6, 119.4, 95.3, 86.2; MS (HRMS) m/z Calcd. for $C_{14}H_9ClNO^+$: 242.0367, $[M+H]^+$ found : 242.0364.



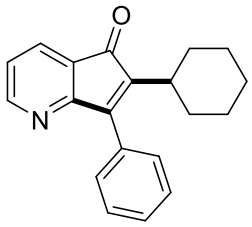
1-([2,3'-bipyridin]-5-yl)-3-phenylprop-2-yn-1-one (1t)

White solid (511.2 mg, 48%); R_f = 0.30 (petroleum ether/ethyl acetate = 1:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.538-9.536 (m, 1H), 9.32 (s, 1H), 8.74 (s, 1H), 8.53 (dd, J_1 = 8.3 Hz, J_2 = 2.2 Hz, 1H), 8.43 (d, J = 8.0 Hz, 1H), 7.93 (d, J = 8.2 Hz, 1H), 7.72 (d, J = 7.0 Hz, 2H), 7.53 (t, J = 7.5 Hz, 1H), 7.48-7.45 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 176.5, 150.3, 149.8, 139.2, 133.3, 132.7, 131.3, 129.8, 129.7, 129.5, 128.9, 127.9, 126.9, 119.8, 94.8, 86.6; MS (HRMS) m/z Calcd. for $C_{19}H_{13}N_2O^+$: 285.1022, $[M+H]^+$ found : 285.1024.



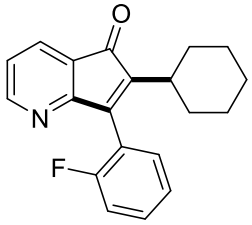
3-phenyl-1-(2-phenylpyridin-3-yl)prop-2-yn-1-one (1u)

Light yellow solid (1.188 g, 42% in total); R_f = 0.32 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.80-8.79 (m, 1H), 8.18 (dd, J_1 = 7.8 Hz, J_2 = 1.7 Hz, 1H), 7.66 (d, J = 8.0 Hz, 2H), 7.45 (t, J = 7.4 Hz, 2H), 7.39 (t, J = 7.4 Hz, 1H), 7.37-7.35 (m, 1H), 7.33 (t, J = 7.5 Hz, 1H), 7.23 (t, J = 7.8 Hz, 2H), 7.16 (d, J = 8.3 Hz, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 179.7, 159.3, 151.9, 139.6, 137.8, 134.1, 133.1, 130.9, 130.1, 129.5, 128.53, 128.45, 122.0, 119.7, 94.9, 88.5; MS (HRMS) m/z Calcd. for $C_{20}H_{14}NO^+$: 284.1070, $[M+H]^+$ found : 284.1073.



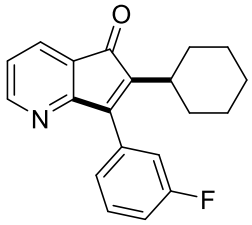
6-cyclohexyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (3a)

Yellow oil (56.3 mg, 78%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.43 (d, *J* = 5.2 Hz, 1H), 7.65 (d, *J* = 7.1 Hz, 1H), 7.57-7.52 (m, 4H), 7.48 (t, *J* = 7.2 Hz, 1H), 7.09-7.07 (m, 1H), 2.70-2.65 (m, 1H), 1.96-1.89 (m, 2H), 1.81-1.77 (m, 2H), 1.68-1.63 (m, 2H), 1.35-1.22 (m, 4H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.2, 167.2, 153.4, 151.9, 142.7, 131.3, 129.5, 129.0, 128.62, 128.55, 125.4, 122.1, 36.4, 31.0, 26.5, 25.8; MS (HRMS) m/z Calcd. for $C_{20}H_{20}NO^+$: 290.1539, $[M+H]^+$ found : 290.1545.



6-cyclohexyl-7-(2-fluorophenyl)-5H-cyclopenta[b]pyridin-5-one (3b)

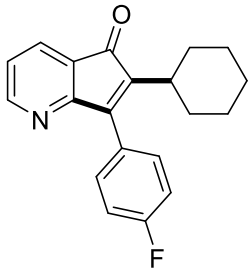
Yellow oil (47.6 mg, 62%); R_f = 0.42 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.41 (d, *J* = 5.2 Hz, 1H), 7.65 (d, *J* = 7.0 Hz, 1H), 7.48-7.45 (m, 1H), 7.42 (t, *J* = 7.3 Hz, 1H), 7.30 (t, *J* = 7.5 Hz, 1H), 7.24 (t, *J* = 9.0 Hz, 1H), 7.09-7.06 (m, 1H), 2.46-2.42 (m, 1H), 1.81-1.75 (m, 4H), 1.67-1.65 (m, 3H), 1.28-1.17 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 195.7, 166.9, 159.7 (d, *J* = 247.4 Hz), 152.3, 148.5, 145.0, 131.2 (d, *J* = 8.1 Hz), 130.7 (d, *J* = 3.1 Hz), 128.8, 124.9, 124.3 (d, *J* = 2.5 Hz), 122.2, 119.5, 116.3 (d, *J* = 21.6 Hz), 37.1, 30.7, 26.5, 25.8. MS (HRMS) m/z Calcd. for $C_{20}H_{19}FNO^+$: 308.1445, $[M+H]^+$ found : 308.1442.



6-cyclohexyl-7-(3-fluorophenyl)-5H-cyclopenta[b]pyridin-5-one (3c)

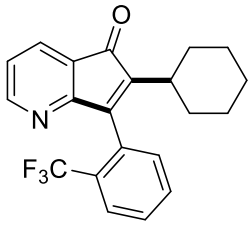
Yellow oil (54.5 mg, 71%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.43 (d, *J* = 5.3 Hz, 1H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.53-7.47 (m, 1H), 7.33-7.29 (m, 2H), 7.20-7.15 (m, 1H), 7.10 (t, *J* = 6.8 Hz, 1H), 2.68-2.60 (m, 1H), 1.95-1.85 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.62 (m, 3H), 1.33-1.22 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.9, 166.8, 162.7 (d, *J* =

245.2 Hz), 152.0, 143.2, 133.3 (d, *J* = 8.1 Hz), 130.2 (d, *J* = 8.4 Hz), 128.9, 125.1, 124.8 (d, *J* = 3.4 Hz), 122.3, 116.4 (d, *J* = 21.0 Hz), 116.1 (d, *J* = 22.3 Hz), 36.5, 31.0, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}FNO^+$: 308.1445, $[M+H]^+$ found : 308.1440.



6-cyclohexyl-7-(4-fluorophenyl)-5H-cyclopenta[b]pyridin-5-one (3d)

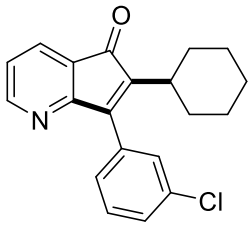
Yellow oil (59.9 mg, 78%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.42 (d, *J* = 5.3 Hz, 1H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.59-7.55 (m, 2H), 7.23 (t, *J* = 8.7 Hz, 2H), 7.11-7.08 (m, 1H), 2.67-2.60 (m, 1H), 1.97-1.88 (m, 2H), 1.81-1.78 (m, 2H), 1.71-1.62 (m, 3H), 1.34-1.21 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.0, 166.9, 163.4 (d, *J* = 248.4 Hz), 152.2, 151.8, 142.6, 131.1 (d, *J* = 8.1 Hz), 128.7, 127.2 (d, *J* = 3.4 Hz), 125.3, 122.3, 115.8 (d, *J* = 21.5 Hz), 36.5, 31.0, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}FNO^+$: 308.1445, $[M+H]^+$ found : 308.1443.



6-cyclohexyl-7-(2-(trifluoromethyl)phenyl)-5H-cyclopenta[b]pyridin-5-one (3e)

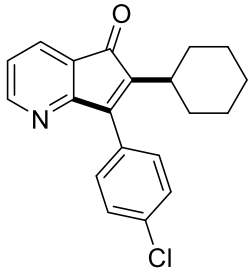
Yellow oil (61.6 mg, 69%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.36 (dd, J_1= 5.3 Hz, J_2= 1.6 Hz, 1H), 7.85 (d, *J* = 7.9 Hz, 1H), 7.69-7.65 (m, 2H), 7.59 (t, *J* = 7.7 Hz, 1H), 7.29 (d, *J* = 7.6 Hz, 1H), 7.08-7.05 (m, 1H), 2.27-2.22 (m, 1H), 1.74-1.68 (m, 3H), 1.67-1.63 (m, 2H), 1.59-1.55 (m, 2H), 1.20-1.10 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.6, 168.0, 152.9, 152.6, 144.5, 132.2, 130.8 (q, *J* = 10.9 Hz), 129.8, 129.1, 128.9, 127.0

(q, *J* = 19.9 Hz), 124.8, 124.2, 123.0, 122.2, 36.8, 31.0, 30.3, 26.44, 26.35, 25.7. MS (HRMS) m/z Calcd. for $C_{21}H_{19}F_3NO^+$: 358.1413, $[M+H]^+$ found : 358.1415.



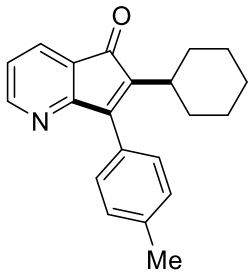
7-(3-chlorophenyl)-6-cyclohexyl-5H-cyclopenta[b]pyridin-5-one (3f)

Yellow oil (56.5 mg, 70%); R_f = 0.42 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.42 (d, *J* = 5.2 Hz, 1H), 7.65 (d, *J* = 7.2 Hz, 1H), 7.55 (s, 1H), 7.47-7.42 (m, 3H), 7.09 (t, *J* = 8.2 Hz, 1H), 2.64-2.60 (m, 1H), 1.92-1.86 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.68 (m, 1H), 1.64-1.62 (m, 2H), 1.32-1.22 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.8, 166.7, 152.0, 151.8, 143.3, 134.6, 133.0, 129.9, 129.6, 129.1, 128.1, 127.2, 125.0, 122.3, 36.5, 31.0, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}ClNO^+$: 324.1150, $[M+H]^+$ found : 324.1153.



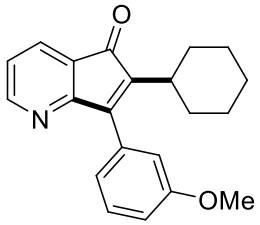
7-(4-chlorophenyl)-6-cyclohexyl-5H-cyclopenta[b]pyridin-5-one (3g)

Yellow oil (59.8 mg, 74%); R_f = 0.43 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.42 (d, *J* = 5.2 Hz, 1H), 7.65 (d, *J* = 7.1 Hz, 1H), 7.510-7.508 (m, 4H), 7.10-7.08 (m, 1H), 2.64-2.60 (m, 1H), 1.94-1.87 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.68 (m, 1H), 1.63-1.61 (m, 2H), 1.30-1.22 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.9, 166.8, 152.1, 151.9, 142.9, 135.6, 130.4, 129.6, 128.9, 128.8, 125.2, 122.3, 36.5, 31.0, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}ClNO^+$: 324.1150, $[M+H]^+$ found : 324.1144.



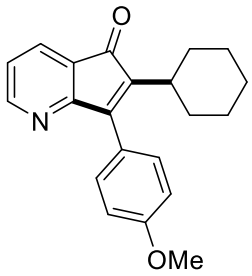
6-cyclohexyl-7-(p-tolyl)-5H-cyclopenta[b]pyridin-5-one (3h)

Yellow oil (60.6 mg, 80%); R_f = 0.42 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.42 (d, *J* = 5.3 Hz, 1H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.48 (d, *J* = 7.9 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.08-7.05 (m, 1H), 2.72-2.65 (m, 1H), 2.44 (s, 3H), 1.98-1.88 (m, 2H), 1.80-1.77 (m, 2H), 1.70-1.62 (m, 3H), 1.31-1.21 (m, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 196.3, 167.2, 153.5, 151.8, 142.1, 139.7, 129.3, 129.0, 128.5, 128.3, 125.5, 122.1, 36.5, 31.0, 26.6, 25.8, 21.7; MS (HRMS) m/z Calcd. for $C_{21}H_{22}NO^+$: 304.1696, $[M+H]^+$ found : 304.1700.



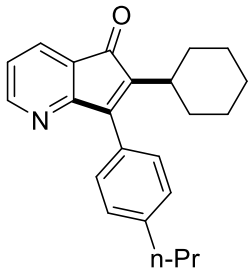
6-cyclohexyl-7-(3-methoxyphenyl)-5H-cyclopenta[b]pyridin-5-one (3i)

Yellow oil (58.2 mg, 73%); R_f = 0.43 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.44-8.42 (m, 1H), 7.64 (d, *J* = 7.2 Hz, 1H), 7.45 (t, *J* = 8.0 Hz, 1H), 7.14 (d, *J* = 7.6 Hz, 1H), 7.10-7.06 (m, 2H), 7.02 (d, *J* = 8.3 Hz, 1H), 3.87 (s, 3H), 2.72-2.65 (m, 1H), 1.96-1.87 (m, 2H), 1.80-1.77 (m, 2H), 1.70-1.62 (m, 3H), 1.34-1.21 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.2, 167.1, 159.6, 153.3, 151.9, 142.8, 132.5, 129.7, 128.6, 125.3, 122.1, 121.3, 115.2, 114.5, 55.3, 36.5, 31.0, 26.5, 25.8; MS (HRMS) m/z Calcd. for $C_{21}H_{22}NO_2^+$: 320.1645, $[M+H]^+$ found : 320.1648.



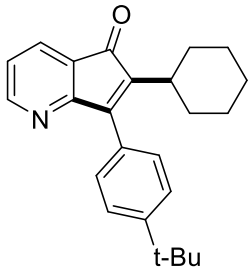
6-cyclohexyl-7-(p-tolyl)-5H-cyclopenta[b]pyridin-5-one (3j)

Yellow solid (59.1 mg, 74%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.43-8.41 (m, 1H), 7.62 (d, *J* = 7.2 Hz, 1H), 7.57 (d, *J* = 8.7 Hz, 2H), 7.08-7.05 (m, 3H), 3.88 (s, 3H), 2.73-2.66 (m, 1H), 2.00-1.91 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.62 (m, 3H), 1.34-1.22 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.3, 167.1, 160.7, 153.0, 151.6, 141.6, 130.7, 128.4, 125.6, 123.5, 122.1, 114.1, 55.4, 36.5, 31.0, 26.6, 25.8; MS (HRMS) m/z Calcd. for $C_{21}H_{22}NO_2^+$: 320.1645, $[M+H]^+$ found : 320.1646.



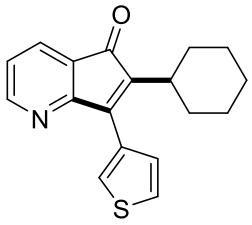
6-cyclohexyl-7-(4-propylphenyl)-5H-cyclopenta[b]pyridin-5-one (3k)

Yellow oil (56.3 mg, 68%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.43-8.41 (m, 1H), 7.63 (d, *J* = 7.2 Hz, 1H), 7.50 (d, *J* = 8.0 Hz, 2H), 7.34 (d, *J* = 7.9 Hz, 2H), 7.07 (t, *J* = 6.2 Hz, 1H), 2.71-2.65 (m, 3H), 1.97-1.91 (m, 2H), 1.80-1.78 (m, 2H), 1.73-1.70 (m, 3H), 1.65-1.63 (m, 2H), 1.29-1.20 (m, 2H), 1.01 (t, *J* = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 196.3, 167.2, 153.4, 151.8, 144.5, 142.2, 129.0, 128.7, 128.49, 128.48, 125.5, 122.1, 38.2, 36.5, 31.0, 26.5, 25.8, 24.4, 14.1; MS (HRMS) m/z Calcd. for $C_{23}H_{26}NO^+$: 332.2009, $[M+H]^+$ found : 332.2016.



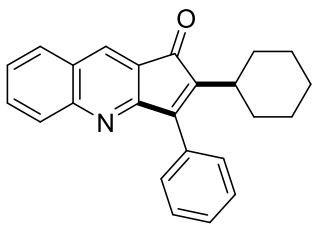
7-(4-(tert-butyl)phenyl)-6-cyclohexyl-5H-cyclopenta[b]pyridin-5-one (3l)

Yellow oil (57.8 mg, 70%); R_f = 0.42 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.44 (dd, J_1 = 5.2 Hz, J_2 = 1.5 Hz, 1H), 7.63 (dd, J_1 = 7.2 Hz, J_2 = 1.6 Hz, 1H), 7.56-7.53 (m, 4H), 7.08-7.06 (m, 1H), 2.74-2.69 (m, 1H), 2.00-1.93 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.68 (m, 1H), 1.67-1.63 (m, 2H), 1.39 (s, 9H), 1.33-1.29 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.3, 167.1, 153.2, 152.7, 151.7, 142.2, 128.8, 128.5, 128.2, 125.59, 125.55, 122.1, 36.5, 35.0, 31.3, 31.0, 26.5, 25.8; MS (HRMS) m/z Calcd. for $C_{24}H_{28}NO^+$: 346.2165, $[M+H]^+$ found : 346.2162.



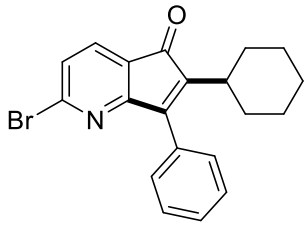
6-cyclohexyl-7-(thiophen-3-yl)-5H-cyclopenta[b]pyridin-5-one (3m)

Yellow oil (53.1 mg, 72%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.46 (d, J = 5.2 Hz, 1H), 8.07 (s, 1H), 7.64 (d, J = 7.2 Hz, 1H), 7.52-7.51 (m, 1H), 7.50-7.49 (m, 1H), 7.11-7.09 (m, 1H), 2.87-2.83 (m, 1H), 2.04-1.97 (m, 2H), 1.85-1.83 (m, 2H), 1.74-1.72 (m, 1H), 1.68-1.66 (m, 2H), 1.36-1.31 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.1, 166.7, 151.6, 147.4, 141.3, 131.7, 128.6, 128.4, 128.3, 125.8, 125.7, 122.2, 37.1, 30.8, 26.7, 25.8; MS (HRMS) m/z Calcd. for $C_{18}H_{18}NOS^+$: 296.1104, $[M+H]^+$ found : 296.1100.



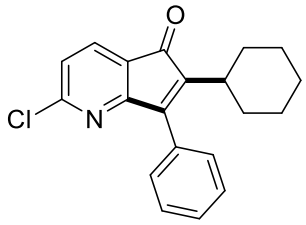
2-cyclohexyl-3-phenyl-1H-cyclopenta[b]quinolin-1-one (3n)

Yellow solid (62.7 mg, 74%); R_f = 0.38 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.93 (s, 1H), 8.03 (d, *J* = 8.5 Hz, 1H), 7.65-7.62 (m, 1H), 7.58-7.56 (m, 3H), 7.39-7.37 (m, 2H), 7.20-7.17 (m, 1H), 7.06 (d, *J* = 8.7 Hz, 1H), 2.39-2.34 (m, 1H), 1.75-1.72 (m, 3H), 1.65-1.58 (m, 5H), 1.23-1.14 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 198.1, 153.51, 153.47, 153.2, 142.3, 141.6, 134.9, 131.5, 130.6, 129.1, 128.9, 128.2, 127.0, 125.0, 122.7, 120.1, 36.0, 31.2, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{24}H_{22}NO^+$: 340.1696, $[M+H]^+$ found : 340.1700.



2-bromo-6-cyclohexyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (3o)

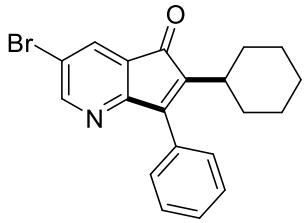
Yellow oil (73.4 mg, 80%); R_f = 0.65 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 7.58 (d, *J* = 7.1 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 2H), 7.50-7.48 (m, 2H), 7.32 (d, *J* = 7.6 Hz, 1H), 2.72-2.67 (m, 1H), 1.96-1.89 (m, 2H), 1.81-1.77 (m, 2H), 1.70-1.63 (m, 3H), 1.34-1.21 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.1, 168.3, 152.1, 145.6, 143.4, 130.5, 130.4, 129.8, 129.2, 128.5, 126.1, 123.9, 36.6, 31.0, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}BrNO^+$: 368.0645, $[M+H]^+$ found : 368.0649.



2-chloro-6-cyclohexyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (3p)

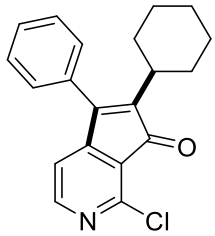
Yellow oil (63.4 mg, 79%); R_f = 0.63 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 7.60-7.48 (m, 6H), 7.14 (d, *J* = 7.6 Hz, 1H), 2.73-2.66 (m, 1H), 1.97-1.88 (m, 2H), 1.81-1.78 (m, 2H), 1.70-1.62 (m, 3H), 1.34-1.21 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 194.9, 168.2, 154.6, 152.1,

143.5, 130.7, 130.5, 129.8, 129.2, 128.6, 123.7, 122.2, 36.6, 30.9, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}ClNO^+$: 324.1150, $[M+H]^+$ found : 324.1153.



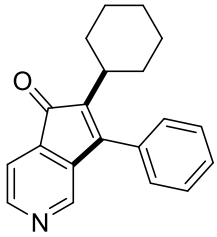
3-bromo-6-cyclohexyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (3q)

Yellow oil (67.8 mg, 76%); R_f = 0.45 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.49 (s, 1H), 7.75 (s, 1H), 7.54-7.53 (m, 4H), 7.51-7.48 (m, 1H), 2.68-2.63 (m, 1H), 1.93-1.86 (m, 2H), 1.80-1.78 (m, 2H), 1.70-1.68 (m, 1H), 1.64-1.62 (m, 2H), 1.30-1.25 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 194.7, 165.1, 153.3, 152.3, 143.0, 131.5, 130.7, 129.8, 128.9, 128.6, 126.7, 119.7, 36.5, 30.9, 26.4, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}BrNO^+$: 368.0645, $[M+H]^+$ found : 368.0649.



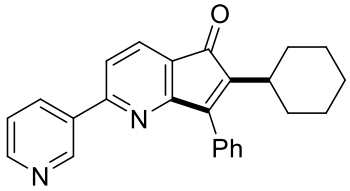
1-chloro-6-cyclohexyl-5-phenyl-7H-cyclopenta[c]pyridin-7-one (3r)

Yellow oil (55.7 mg, 69%); R_f = 0.32 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.54-8.53 (m, 1H), 7.62 (d, *J* = 7.6 Hz, 1H), 7.48 (d, *J* = 7.0 Hz, 1H), 7.42-7.39 (m, 1H), 7.30-7.28 (m, 1H), 7.21 (t, *J* = 7.0 Hz, 1H), 6.61 (d, *J* = 7.1 Hz, 1H), 2.32-2.23 (m, 1H), 1.72-1.63 (m, 5H), 1.31-1.24 (m, 2H), 1.21-1.14 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 194.0, 157.7, 155.5, 150.9, 145.0, 142.3, 131.6, 129.7, 129.2, 127.9, 120.8, 115.1, 36.3, 30.9, 26.4, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{19}ClNO^+$: 324.1150, $[M+H]^+$ found : 324.1154.



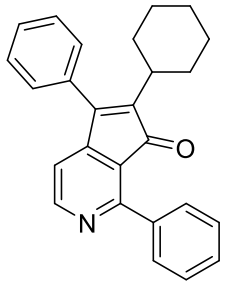
6-cyclohexyl-7-phenyl-5H-cyclopenta[c]pyridin-5-one (3s)

Yellow oil (56.4 mg, 78%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.67 (s, 1H), 8.26 (s, 1H), 7.56 (t, J = 7.3 Hz, 2H), 7.51 (t, J = 7.4 Hz, 1H), 7.43 (d, J = 6.9 Hz, 2H), 7.33-7.32 (m, 1H), 2.57-2.52 (m, 1H), 1.89-1.84 (m, 2H), 1.78-1.75 (m, 2H), 1.68-1.65 (m, 1H), 1.60-1.58 (m, 2H), 1.29-1.19 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 197.2, 155.4, 152.1, 140.4, 140.0, 137.7, 132.3, 129.7, 129.1, 127.8, 115.7, 36.0, 31.0, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{20}H_{20}NO^+$: 290.1539, $[M+H]^+$ found : 290.1543.



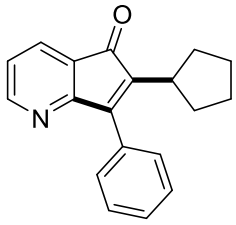
6-cyclohexyl-7-phenyl-2-(pyridin-3-yl)-5H-cyclopenta[b]pyridin-5-one (3t)

White solid (73.2 mg, 80%); R_f = 0.30 (petroleum ether/ethyl acetate = 3:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 9.24 (s, 1H), 8.64-8.63 (m, 1H), 8.29 (td, J_1 = 8.0 Hz, J_2 = 2.0 Hz, 1H), 7.74 (d, J = 7.5 Hz, 1H), 7.69-7.67 (m, 2H), 7.58-7.55 (m, 3H), 7.52-7.49 (m, 1H), 7.38-7.36 (m, 1H), 2.78-2.73 (m, 1H), 2.01-1.95 (m, 2H), 1.82-1.80 (m, 3H), 1.71-1.66 (m, 2H), 1.36-1.31 (m, 1H), 1.29-1.23 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.8, 167.6, 156.8, 152.6, 150.4, 148.5, 143.6, 134.4, 134.2, 131.1, 129.6, 129.44, 129.42, 128.4, 124.4, 123.6, 118.3, 36.7, 31.0, 26.5, 25.8; MS (HRMS) m/z Calcd. for $C_{25}H_{23}N_2O^+$: 367.1805, $[M+H]^+$ found : 367.1806.



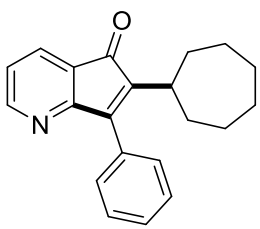
6-cyclohexyl-1,5-diphenyl-7H-cyclopenta[c]pyridin-7-one (3u)

Yellow solid (59.3 mg, 65%); R_f = 0.34 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.64 (d, *J* = 4.6 Hz, 1H), 7.87-7.86 (m, 2H), 7.56 (t, *J* = 7.2 Hz, 2H), 7.52-7.48 (m, 4H), 7.39 (d, *J* = 8.4 Hz, 2H), 6.89 (d, *J* = 4.7 Hz, 1H), 2.59-2.54 (m, 1H), 1.87-1.82 (m, 2H), 1.75-1.73 (m, 2H), 1.66-1.63 (m, 1H), 1.60-1.58 (m, 2H), 1.24-1.16 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.5, 155.9, 155.0, 154.4, 151.6, 141.5, 136.9, 132.1, 129.7, 129.4, 129.0, 128.1, 127.9, 120.6, 114.5, 36.3, 30.9, 26.5, 25.7; MS (HRMS) m/z Calcd. for $C_{26}H_{24}NO^+$: 366.1852, $[M+H]^+$ found : 366.1853.



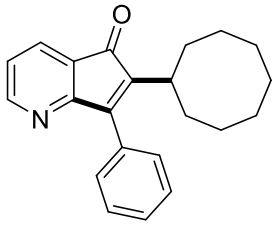
6-cyclopentyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4a)

Yellow oil (55.0 mg, 80%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.43 (d, *J* = 5.2 Hz, 1H), 7.64 (d, *J* = 7.3 Hz, 1H), 7.60 (d, *J* = 7.3 Hz, 2H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.47 (t, *J* = 7.3 Hz, 1H), 7.09-7.06 (m, 1H), 3.07-3.01 (m, 1H), 1.97-1.89 (m, 4H), 1.84-1.80 (m, 2H), 1.62-1.59 (m, 2H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.1, 167.0, 153.9, 151.9, 141.6, 131.2, 129.5, 129.1, 128.6, 128.5, 125.6, 122.1, 36.7, 32.4, 26.6; MS (HRMS) m/z Calcd. for $C_{19}H_{18}NO^+$: 276.1383, $[M+H]^+$ found : 276.1387.



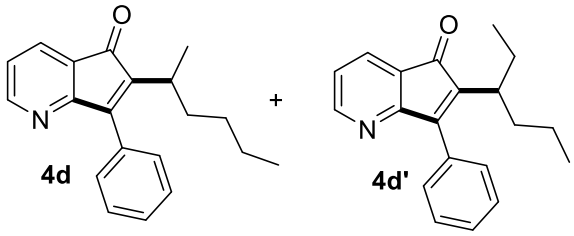
6-cycloheptyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4b)

Yellow oil (56.1 mg, 74%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.43-8.42 (m, 1H), 7.64 (d, *J* = 7.2 Hz, 1H), 7.58-7.56 (m, 2H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.09-7.07 (m, 1H), 2.83-2.78 (m, 1H), 2.02-1.95 (m, 2H), 1.82-1.78 (m, 2H), 1.72-1.68 (m, 2H), 1.61-1.57 (m, 4H), 1.46-1.41 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 196.3, 167.4, 151.8, 151.7, 144.4, 131.3, 129.5, 129.1, 128.6, 128.5, 125.3, 122.1, 37.7, 33.4, 27.9, 27.8; MS (HRMS) m/z Calcd. for $C_{21}H_{22}NO^+$: 304.1696, $[M+H]^+$ found : 304.1701.



6-cyclooctyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4c)

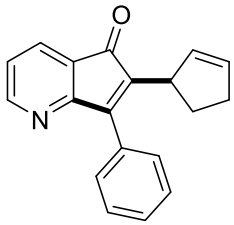
Yellow oil (57.9 mg, 73%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.42 (d, *J* = 5.2 Hz, 1H), 7.64 (d, *J* = 7.2 Hz, 1H), 7.58 (d, *J* = 8.5 Hz, 2H), 7.53 (t, *J* = 7.6 Hz, 2H), 7.47 (t, *J* = 7.4 Hz, 1H), 7.09-7.07 (m, 1H), 2.95-2.90 (m, 1H), 2.04-1.99 (m, 2H), 1.80-1.75 (m, 2H), 1.66-1.61 (m, 3H), 1.57-1.53 (m, 4H), 1.48-1.44 (m, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 196.3, 167.4, 151.8, 151.5, 145.2, 131.2, 129.5, 129.0, 128.6, 128.5, 125.3, 122.1, 35.1, 32.3, 26.5, 26.4, 26.3; MS (HRMS) m/z Calcd. for $C_{21}H_{22}NO^+$: 318.1852, $[M+H]^+$ found : 318.1856.



(S)-6-(hexan-2-yl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one(4d)/(S)-6-(hexan-3-yl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4d')

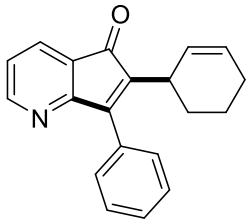
Yellow oil (50.9 mg, 70%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.43 (d, *J* = 5.1 Hz, 1H), 7.65 (d, *J* = 7.1 Hz, 1H),

7.55-7.51 (m, 4H), 7.47-7.45 (m, 1H), 7.10-7.08 (m, 1H), 2.88-2.82 (m, 0.6H), 2.68-2.63 (m, 0.4H), 1.84-1.76 (m, 2.5H), 1.68-1.63 (m, 0.5H), 1.61-1.53 (m, 1.3H), 1.30 (d, J = 7.0 Hz, 1.8H), 1.22-1.15 (m, 2.9H), 0.86 (t, J = 7.5 Hz, 1.8H), 0.81 (t, J = 6.9 Hz, 1.2H), 0.79 (t, J = 7.4 Hz, 1.2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 196.4, 196.2, 167.3, 167.2, 155.9, 154.2, 152.0, 151.9, 142.7, 141.4, 131.3, 131.2, 131.0, 129.5, 129.3, 128.93, 128.86, 128.74, 128.65, 128.64, 128.54, 128.53, 125.4, 125.3, 122.2, 38.5, 36.0, 34.9, 31.3, 30.5, 27.0, 22.7, 21.5, 19.8, 14.2, 14.1, 12.9; MS (HRMS) m/z Calcd. for $C_{20}H_{22}NO^+$: 292.1696, $[M+H]^+$ found : 292.1690.



(R)-6-(cyclopent-2-en-1-yl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4e)

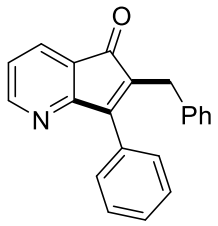
Yellow oil (45.9 mg, 67%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v);^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.44 (dd, J_1 = 5.3 Hz, J_2 = 1.6 Hz, 1H), 7.65 (dd, J_1 = 7.2 Hz, J_2 = 1.5 Hz, 1H), 7.61 (d, J = 8.5 Hz, 2H), 7.52 (t, J = 7.5 Hz, 2H), 7.47 (t, J = 7.4 Hz, 1H), 7.11-7.08 (m, 1H), 5.89-5.87 (m, 1H), 5.61-5.59 (m, 1H), 4.00-3.97 (m, 1H), 2.69-2.63 (m, 1H), 2.43-2.39 (m, 1H), 2.23-2.18 (m, 1H), 2.05-2.00 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.8, 167.1, 154.1, 151.9, 140.1, 132.4, 131.7, 131.0, 129.7, 129.3, 128.7, 128.4, 125.4, 122.3, 41.9, 33.0, 30.0; MS (HRMS) m/z Calcd. for $C_{19}H_{16}NO^+$: 274.1226, $[M+H]^+$ found : 274.1223.



(R)-6-(cyclohex-2-en-1-yl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4f)

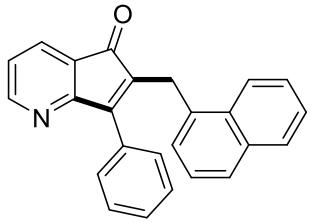
Yellow oil (48.8 mg, 68%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v);^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.44 (dd, J_1 = 5.2 Hz, J_2 = 1.6 Hz, 1H), 7.67 (dd, J_1 = 7.2 Hz, J_2 = 1.6 Hz, 1H), 7.59 (d, J = 7.1 Hz, 2H), 7.51 (t, J = 7.4 Hz, 2H), 7.46 (t, J = 8.0 Hz, 1H), 7.11-7.09 (m, 1H), 5.79-5.75 (m, 1H), 5.50-5.48 (m, 1H), 3.57-3.52

(m, 1H), 2.17-2.12 (m, 1H), 2.06-2.01 (m, 1H), 1.97-1.92 (m, 1H), 1.89-1.83 (m, 2H), 1.61-1.56 (m, 1H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.8, 167.2, 154.5, 151.9, 141.2, 131.0, 129.7, 129.2, 128.8, 128.4, 128.2, 128.0, 125.3, 122.3, 33.3, 28.4, 24.7, 22.5; MS (HRMS) m/z Calcd. for $C_{20}H_{18}NO^+$: 288.1383, $[M+H]^+$ found : 288.1380.



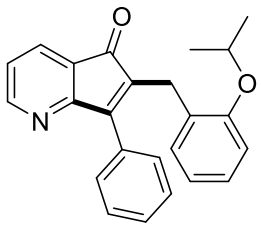
6-benzyl-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4g)

Yellow oil (8.9 mg, 12%); R_f = 0.38 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.50-8.49 (m, 1H), 7.72 (dd, J_1 = 7.2 Hz, J_2 = 1.6 Hz, 1H), 7.70-7.68 (m, 2H), 7.53-7.48 (m, 3H), 7.33-7.30 (m, 1H), 7.27-7.26 (m, 1H), 7.21-7.19 (m, 3H), 7.15-7.13 (m, 1H), 3.87 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 195.9, 167.4, 154.8, 151.9, 138.8, 136.7, 130.9, 130.0, 129.11, 129.06, 128.71, 128.66, 128.5, 126.4, 125.3, 122.4, 29.3; MS (HRMS) m/z Calcd. for $C_{21}H_{16}NO^+$: 298.1226, $[M+H]^+$ found : 298.1224.



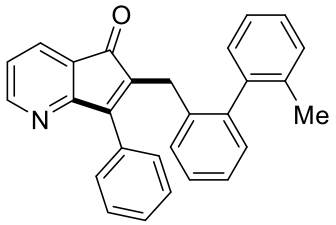
6-(naphthalen-1-ylmethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4h)

Yellow oil (22.6 mg, 26%); R_f = 0.39 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.53 (dd, J_1 = 5.3 Hz, J_2 = 1.3 Hz, 1H), 7.92 (d, J = 8.2 Hz, 1H), 7.87 (d, J = 7.9 Hz, 1H), 7.75 (d, J = 7.3 Hz, 2H), 7.70-7.68 (m, 2H), 7.50 (t, J = 6.8 Hz, 1H), 7.47 (t, J = 7.6 Hz, 1H), 7.44-7.43 (m, 3H), 7.36 (t, J = 7.7 Hz, 1H), 7.28-7.27 (m, 1H), 7.18-7.16 (m, 1H), 4.31 (s, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 195.8, 167.3, 155.5, 151.9, 136.2, 134.4, 133.9, 131.9, 130.8, 130.2, 129.2, 129.0, 128.8, 128.7, 127.3, 126.1, 125.8, 125.63, 125.60, 125.5, 123.6, 122.4, 26.7; MS (HRMS) m/z Calcd. for $C_{25}H_{18}NO^+$: 348.1383, $[M+H]^+$ found : 348.1387.



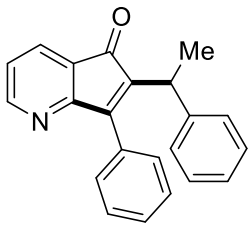
6-(2-isopropoxybenzyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4i)

Yellow oil (30.2 mg, 35%); R_f = 0.38 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.49 (dd, J_1 = 5.3 Hz, J_2 = 1.6 Hz, 1H), 7.71 (dd, J_1 = 7.1 Hz, J_2 = 1.6 Hz, 1H), 7.69-7.67 (m, 2H), 7.46-7.42 (m, 3H), 7.16-7.12 (m, 2H), 7.07 (d, J = 7.6 Hz, 1H), 6.84 (d, J = 8.2 Hz, 1H), 6.80 (d, J = 7.7 Hz, 1H), 4.57-4.53 (m, 1H), 3.85 (s, 2H), 1.26 (d, J = 6.0 Hz, 6H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.9, 167.5, 155.5, 151.8, 137.1, 131.0, 129.8, 129.4, 129.3, 129.2, 128.9, 128.5, 128.0, 127.4, 125.6, 122.2, 120.2, 112.8, 70.1, 24.1, 22.2; MS (HRMS) m/z Calcd. for $C_{24}H_{22}NO_2^+$: 356.1645, $[M+H]^+$ found : 356.1648.



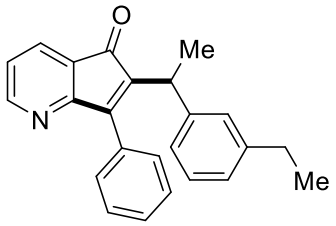
6-((2'-methyl-[1,1'-biphenyl]-2-yl)methyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4j)

Yellow oil (54.2 mg, 56%); R_f = 0.37 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.49 (dd, J_1 = 5.3 Hz, J_2 = 1.6 Hz, 1H), 7.68 (dd, J_1 = 7.2 Hz, J_2 = 1.6 Hz, 1H), 7.62-7.61 (m, 2H), 7.45-7.41 (m, 3H), 7.26-7.25 (m, 1H), 7.23-7.22 (m, 2H), 7.20-7.19 (m, 1H), 7.18-7.17 (m, 2H), 7.16-7.14 (m, 2H), 7.13-7.12 (m, 1H), 3.62 (d, J = 16.3 Hz, 1H), 3.53 (d, J = 16.2 Hz, 1H), 2.11 (s, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.4, 167.2, 154.9, 151.8, 141.4, 140.9, 136.60, 136.59, 136.1, 130.7, 130.1, 130.0, 129.8, 129.6, 129.03, 129.0, 128.5, 127.9, 127.6, 127.5, 126.4, 125.7, 125.4, 27.6, 20.0; MS (HRMS) m/z Calcd. for $C_{28}H_{22}NO^+$: 388.1696, $[M+H]^+$ found : 388.1692.



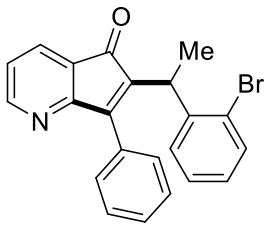
(S)-7-phenyl-6-(1-phenylethyl)-5H-cyclopenta[b]pyridin-5-one (4k)

Yellow oil (55.2 mg, 71%); R_f = 0.38 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.46 (d, *J* = 5.2 Hz, 1H), 7.65 (d, *J* = 7.2 Hz, 1H), 7.57 (d, *J* = 8.1 Hz, 2H), 7.53 (t, *J* = 7.4 Hz, 2H), 7.50-7.48 (m, 1H), 7.33 (d, *J* = 7.4 Hz, 2H), 7.29 (t, *J* = 7.7 Hz, 2H), 7.20 (t, *J* = 7.2 Hz, 1H), 7.12-7.10 (m, 1H), 4.23 (q, *J* = 7.2 Hz, 1H), 1.70 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.8, 167.0, 154.5, 152.0, 143.5, 141.0, 130.9, 129.8, 129.0, 128.9, 128.7, 128.5, 127.7, 126.4, 125.2, 122.5, 36.0, 19.2; MS (HRMS) m/z Calcd. for $C_{22}H_{18}NO^+$: 312.1383, $[M+H]^+$ found : 312.1388.



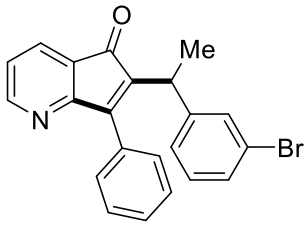
(S)-6-(1-(3-ethylphenyl)ethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4l)

Yellow oil (60.2 mg, 71%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.45 (d, *J* = 5.1 Hz, 1H), 7.66 (d, *J* = 7.2 Hz, 1H), 7.58 (d, *J* = 7.0 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 2H), 7.50 (t, *J* = 7.3 Hz, 1H), 7.22 (t, *J* = 7.8 Hz, 1H), 7.17-7.15 (m, 2H), 7.11-7.09 (m, 1H), 7.05 (d, *J* = 7.4 Hz, 1H), 4.23 (q, *J* = 7.2 Hz, 1H), 2.64 (q, *J* = 7.6 Hz, 2H), 1.70 (d, *J* = 7.3 Hz, 3H), 1.24 (t, *J* = 7.6 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.9, 167.1, 154.5, 152.0, 144.4, 143.5, 141.2, 131.0, 129.7, 129.0, 128.9, 128.6, 128.5, 127.3, 126.0, 125.2, 125.0, 122.4, 36.1, 29.0, 19.3, 15.7; MS (HRMS) m/z Calcd. for $C_{24}H_{22}NO^+$: 340.1696, $[M+H]^+$ found : 340.1699.



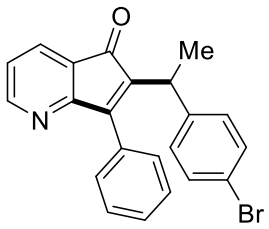
(R)-6-(1-(2-bromophenyl)ethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4m)

Yellow oil (63.2 mg, 65%); R_f = 0.38 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.45 (d, *J* = 5.2 Hz, 1H), 7.66 (d, *J* = 7.3 Hz, 1H), 7.58 (d, *J* = 7.9 Hz, 1H), 7.48-7.46 (m, 4H), 7.45-7.42 (m, 2H), 7.24 (t, *J* = 7.5 Hz, 1H), 7.12-7.10 (m, 1H), 7.03 (t, *J* = 7.6 Hz, 1H), 4.50 (q, *J* = 7.2 Hz, 1H), 1.73 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.9, 166.9, 155.4, 152.1, 142.2, 139.3, 132.8, 131.1, 129.9, 129.7, 128.93, 128.9, 128.5, 128.0, 127.4, 125.1, 124.4, 122.5, 36.1, 19.7; MS (HRMS) m/z Calcd. for $C_{22}H_{17}BrNO^+$: 390.0488, $[M+H]^+$ found : 390.0494.



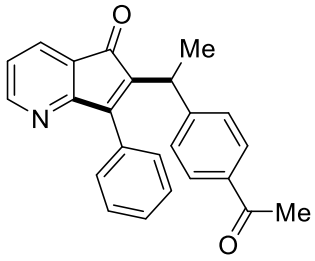
(S)-6-(1-(3-bromophenyl)ethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4n)

Yellow oil (68.1 mg, 70%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.47 (d, *J* = 5.2 Hz, 1H), 7.66 (d, *J* = 7.1 Hz, 1H), 7.55-7.52 (m, 4H), 7.51-7.50 (m, 1H), 7.45 (s, 1H), 7.32 (d, *J* = 7.9 Hz, 1H), 7.24 (d, *J* = 7.8 Hz, 1H), 7.16-7.11 (m, 2H), 4.20 (q, *J* = 7.2 Hz, 1H), 1.68 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.6, 166.8, 154.9, 152.2, 145.8, 140.1, 130.8, 130.7, 130.0, 129.9, 129.6, 129.1, 128.9, 128.7, 126.4, 125.1, 122.64, 122.6, 35.7, 19.1; MS (HRMS) m/z Calcd. for $C_{22}H_{17}BrNO^+$: 390.0488, $[M+H]^+$ found : 390.0490.



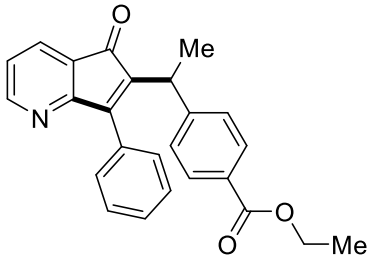
(S)-6-(1-(4-bromophenyl)ethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4o)

Yellow oil (67.1 mg, 69%); R_f = 0.40 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.46 (d, *J* = 5.4 Hz, 1H), 7.65 (d, *J* = 7.4 Hz, 1H), 7.56-7.49 (m, 5H), 7.40 (d, *J* = 8.6 Hz, 2H), 7.19 (d, *J* = 8.8 Hz, 2H), 7.11 (t, *J* = 6.3 Hz, 1H), 4.18 (q, *J* = 7.9 Hz, 1H), 1.68 (d, *J* = 7.5 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 195.7, 166.8, 154.8, 152.2, 142.5, 140.3, 131.6, 130.8, 129.9, 129.5, 129.0, 128.9, 128.7, 125.1, 122.6, 120.3, 35.6, 19.1; MS (HRMS) m/z Calcd. for $C_{22}H_{17}BrNO^+$: 390.0488, $[M+H]^+$ found : 390.0490.



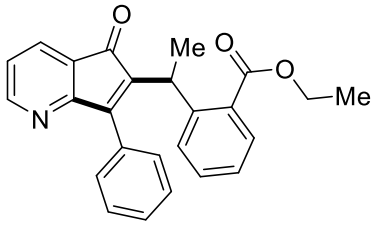
(S)-6-(1-(4-acetylphenyl)ethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4p)

Yellow oil (61.8 mg, 70%); R_f = 0.34 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.47 (d, *J* = 5.2 Hz, 1H), 7.88 (d, *J* = 8.3 Hz, 2H), 7.65 (d, *J* = 7.2 Hz, 1H), 7.57-7.50 (m, 5H), 7.40 (d, *J* = 8.3 Hz, 2H), 7.13-7.11 (m, 1H), 4.28 (q, *J* = 7.2 Hz, 1H), 2.57 (s, 3H), 1.72 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 197.8, 195.6, 166.8, 155.1, 152.2, 149.0, 140.0, 135.5, 130.7, 130.0, 129.0, 128.9, 128.8, 128.7, 127.9, 125.1, 122.6, 36.0, 26.6, 19.0; MS (HRMS) m/z Calcd. for $C_{24}H_{20}NO_2^+$: 354.1489, $[M+H]^+$ found : 354.1492.



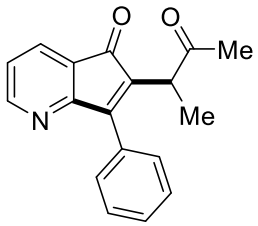
ethyl (S)-4-(1-(5-oxo-7-phenyl-5H-cyclopenta[b]pyridin-6-yl)ethyl)benzoate (4q)

Yellow oil (77.6 mg, 81%); R_f = 0.38 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.47 (d, *J* = 5.2 Hz, 1H), 7.97 (d, *J* = 8.2 Hz, 2H), 7.66 (d, *J* = 7.3 Hz, 1H), 7.56-7.50 (m, 5H), 7.38 (d, *J* = 8.3 Hz, 2H), 7.13-7.11 (m, 1H), 4.36 (q, *J* = 7.1 Hz, 2H), 4.27 (q, *J* = 7.2 Hz, 1H), 1.71 (d, *J* = 7.2 Hz, 3H), 1.38 (t, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 195.6, 166.8, 166.6, 155.0, 152.2, 148.7, 140.1, 130.7, 130.0, 129.8, 129.0, 128.9, 128.75, 128.71, 127.6, 125.1, 122.6, 60.9, 36.0, 19.0, 14.4; MS (HRMS) m/z Calcd. for $C_{25}H_{22}NO_3^+$: 384.1594, $[M+H]^+$ found : 384.1589.



ethyl (S)-2-(1-(5-oxo-7-phenyl-5H-cyclopenta[b]pyridin-6-yl)ethyl)benzoate (4r)

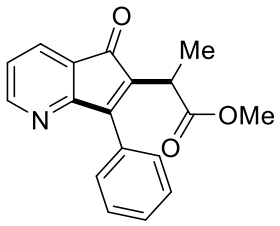
Yellow oil (46.8 mg, 53%); R_f = 0.37 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.44-8.43 (m, 1H), 7.71-7.68 (m, 2H), 7.60 (d, *J* = 7.8 Hz, 1H), 7.40-7.37 (m, 4H), 7.36-7.33 (m, 2H), 7.21 (d, *J* = 7.7 Hz, 1H), 7.14-7.11 (m, 1H), 5.00 (q, *J* = 7.2 Hz, 1H), 4.11 (q, *J* = 7.2 Hz, 2H), 1.69 (d, *J* = 7.2 Hz, 3H), 1.23 (t, *J* = 7.1 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 196.3, 168.1, 166.9, 151.9, 144.5, 141.5, 131.7, 130.4, 130.2, 129.5, 129.1, 128.9, 128.4, 126.2, 125.2, 122.6, 61.0, 33.3, 20.5, 14.2; MS (HRMS) m/z Calcd. for $C_{25}H_{22}NO_3^+$: 384.1594, $[M+H]^+$ found : 384.1592.



(S)-6-(3-oxobutan-2-yl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4s)

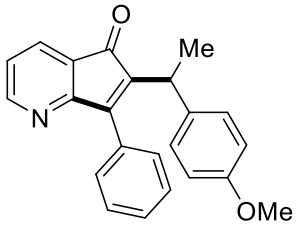
Yellow oil (38.8 mg, 56%); R_f = 0.33 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.50 (d, *J* = 5.2 Hz, 1H), 7.73 (d, *J* = 7.3 Hz, 1H), 7.62 (d, *J* = 7.0 Hz, 2H), 7.54 (t, *J* = 7.3 Hz, 2H), 7.52-7.49 (m, 1H), 7.17-7.15 (m,

1H), 3.70 (q, *J* = 7.1 Hz, 1H), 2.15 (s, 3H), 1.45 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 206.9, 195.1, 166.9, 155.7, 152.2, 137.2, 130.3, 129.4, 129.3, 128.8, 128.7, 122.7, 44.2, 28.4, 15.5; MS (HRMS) m/z Calcd. for $C_{18}H_{16}NO_2^+$: 278.1176, $[M+H]^+$ found : 278.1174.



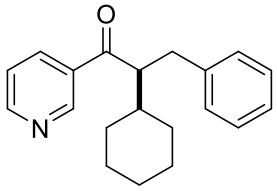
methyl (R)-2-(5-oxo-7-phenyl-5H-cyclopenta[b]pyridin-6-yl)propanoate (4t)

Yellow oil (39.6 mg, 54%); R_f = 0.31 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 600 MHz) δ (ppm): 8.50-8.48 (m, 1H), 7.72 (dd, J_1 = 7.2 Hz, J_2 = 1.5 Hz, 1H), 7.64 (d, *J* = 7.0 Hz, 2H), 7.54 (t, *J* = 7.4 Hz, 2H), 7.50 (t, *J* = 7.4 Hz, 1H), 7.16-7.14 (m, 1H), 3.80 (q, *J* = 7.2 Hz, 1H), 3.64 (s, 3H), 1.49 (d, *J* = 7.2 Hz, 3H); ^{13}C NMR ($CDCl_3$, 150 MHz) δ (ppm): 194.7, 173.1, 166.9, 154.6, 152.1, 136.9, 130.4, 130.1, 129.3, 128.9, 128.8, 125.1, 122.6, 52.4, 35.6, 16.3; MS (HRMS) m/z Calcd. for $C_{18}H_{16}NO_3^+$: 294.1125, $[M+H]^+$ found : 294.1128.



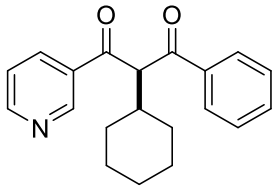
(S)-6-(1-(4-methoxyphenyl)ethyl)-7-phenyl-5H-cyclopenta[b]pyridin-5-one (4u)

Yellow oil (62.4 mg, 73%); R_f = 0.41 (petroleum ether/ethyl acetate = 10:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.45 (dd, J_1 = 5.3 Hz, J_2 = 1.6 Hz, 1H), 7.63 (dd, J_1 = 7.2 Hz, J_2 = 1.6 Hz, 1H), 7.59-7.56 (m, 2H), 7.55-7.53 (m, 2H), 7.52-7.50 (m, 1H), 7.24 (d, *J* = 8.4 Hz, 2H), 7.11-7.08 (m, 1H), 6.82 (d, *J* = 8.8 Hz, 2H), 4.18 (q, *J* = 7.2 Hz, 1H), 3.78 (s, 3H), 1.67 (d, *J* = 7.3 Hz, 3H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 195.9, 167.0, 158.1, 154.1, 152.0, 141.2, 135.6, 131.0, 129.7, 129.0, 128.8, 128.7, 128.6, 125.2, 122.4, 113.9, 55.3, 35.3, 19.3; MS (HRMS) m/z Calcd. for $C_{23}H_{20}NO_2^+$: 342.1489, $[M+H]^+$ found : 342.1483.



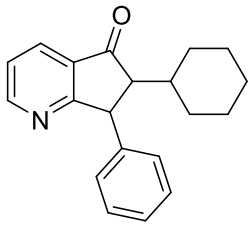
(S)-2-cyclohexyl-3-phenyl-1-(pyridin-3-yl)propan-1-one (9)

White solid (40.3 mg, 56%); R_f = 0.43 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 9.08 (s, 1H), 8.74-8.73 (m, 1H), 8.14-8.11 (m, 1H), 7.41-7.38 (m, 1H), 7.24 (t, *J* = 7.6 Hz, 2H), 7.17-7.13 (m, 3H), 3.43-3.78 (m, 1H), 3.36-3.30 (m, 1H), 3.18-3.12 (m, 1H), 1.89-1.86 (m, 1H), 1.79-1.75 (m, 1H), 1.66-1.55 (m, 3H), 1.50-1.47 (m, 1H), 1.28-1.23 (m, 1H), 1.15-1.09 (m, 2H), 1.04-1.00 (m, 1H), 0.90-0.81 (m, 1H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 198.3, 152.5, 148.9, 143.2, 135.9, 132.8, 128.3, 128.2, 126.3, 123.8, 47.3, 43.0, 42.8, 31.6, 30.9, 26.5, 26.3; MS (HRMS) m/z Calcd. for $C_{20}H_{24}NO^+$: 294.1852, $[M+H]^+$ found : 294.1849.



(R)-2-cyclohexyl-1-phenyl-3-(pyridin-3-yl)propane-1,3-dione (10)

White solid (22.3 mg, 29%); R_f = 0.30 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 9.21 (s, 1H), 8.76-8.75 (m, 1H), 8.30-8.27 (m, 1H), 8.01 (t, *J* = 8.2 Hz, 2H), 7.58 (d, *J* = 7.4 Hz, 1H), 7.47 (t, *J* = 7.7 Hz, 2H), 7.42-7.39 (m, 1H), 5.13 (d, *J* = 9.4 Hz, 1H), 2.72-2.63 (m, 1H), 1.72-1.66 (m, 4H), 1.40-1.26 (m, 4H), 1.16-1.05 (m, 2H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 195.2, 194.5, 153.5, 149.8, 137.1, 136.4, 133.9, 132.6, 129.1, 128.8, 123.9, 64.6, 39.9, 31.9, 31.8, 26.3, 26.2; MS (HRMS) m/z Calcd. for $C_{20}H_{22}NO_2^+$: 308.1645, $[M+H]^+$ found : 308.1640.



6-cyclohexyl-7-phenyl-6,7-dihydro-5H-cyclopenta[b]pyridin-5-one (11)

Colorless oil (66.9 mg, 92%); R_f = 0.34 (petroleum ether/ethyl acetate = 4:1, v/v); ^{1}H NMR ($CDCl_3$, 400 MHz) δ (ppm): 8.79 (d, *J* = 4.7 Hz, 1H), 8.05 (d, *J* = 7.7 Hz, 1H), 7.36-7.33 (m, 1H), 7.30 (d, *J* = 7.6 Hz, 2H), 7.24 (t, *J* = 7.3 Hz, 1H), 7.12 (t, *J* = 7.2 Hz, 2H), 4.50 (d, *J* = 3.5 Hz, 1H), 2.83 (t, *J* = 3.9 Hz, 1H), 2.19-2.11 (m, 1H), 1.75-1.63 (m, 4H), 1.52-1.49 (m, 1H), 1.35-1.21 (m, 5H); ^{13}C NMR ($CDCl_3$, 100 MHz) δ (ppm): 206.7, 175.4, 156.4, 142.7, 131.7, 130.3, 129.1, 128.1, 127.1, 123.1, 63.2, 50.5, 40.6, 31.0, 29.1, 26.6, 26.4, 26.3. MS (HRMS) m/z Calcd. for $C_{20}H_{22}NO^+$: 292.1696, $[M+H]^+$ found : 292.1692.

9. References

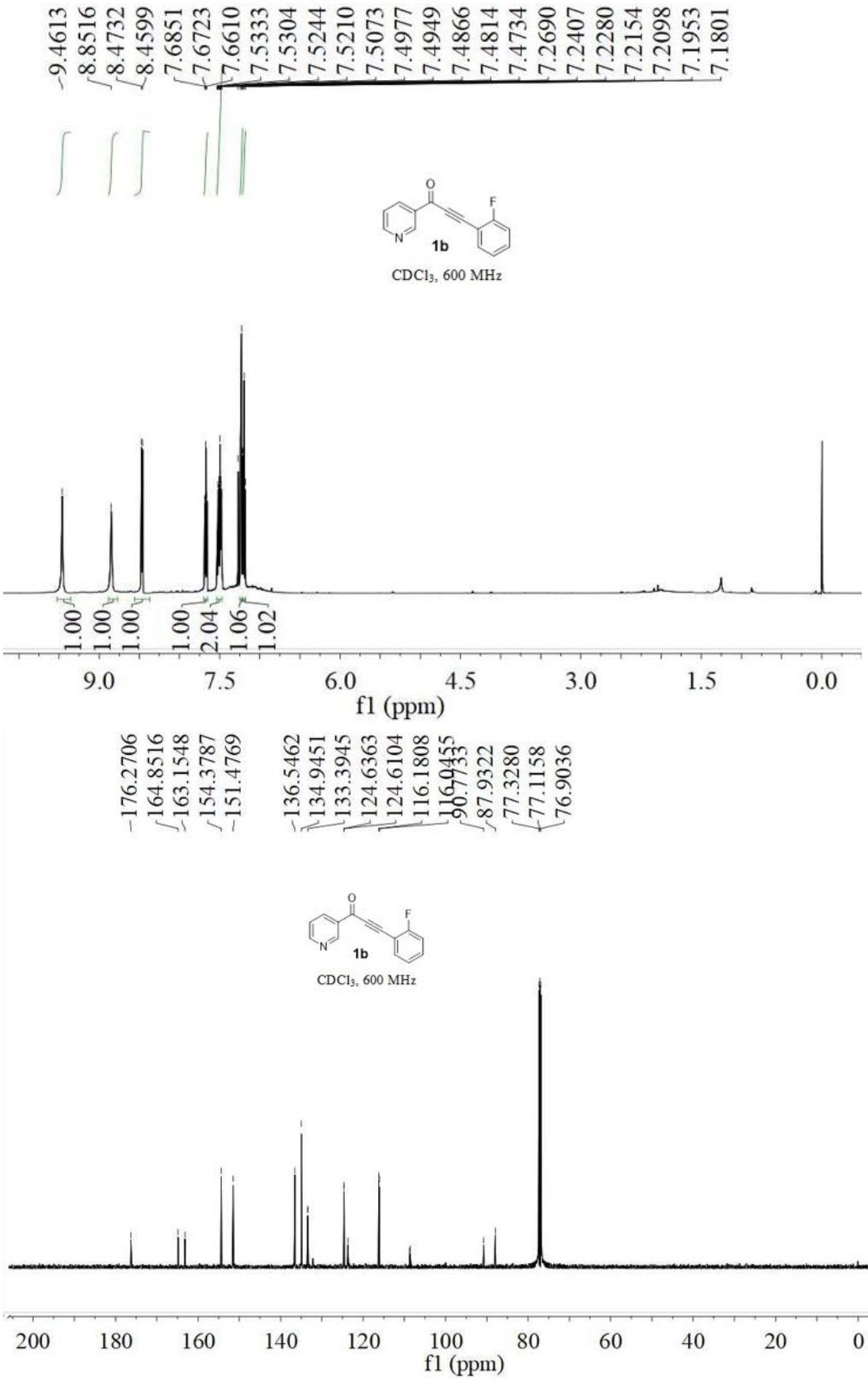
[1] Boersch, C.; Merkul, Eugen.; Muller. T. J. J. Catalytic Syntheses of N-Heterocyclic Ynones and Ynediones by In Situ Activation of Carboxylic Acids with Oxalyl Chloride. *Angew. Chem. Int. Ed.* **2011**, *50*, 10448.

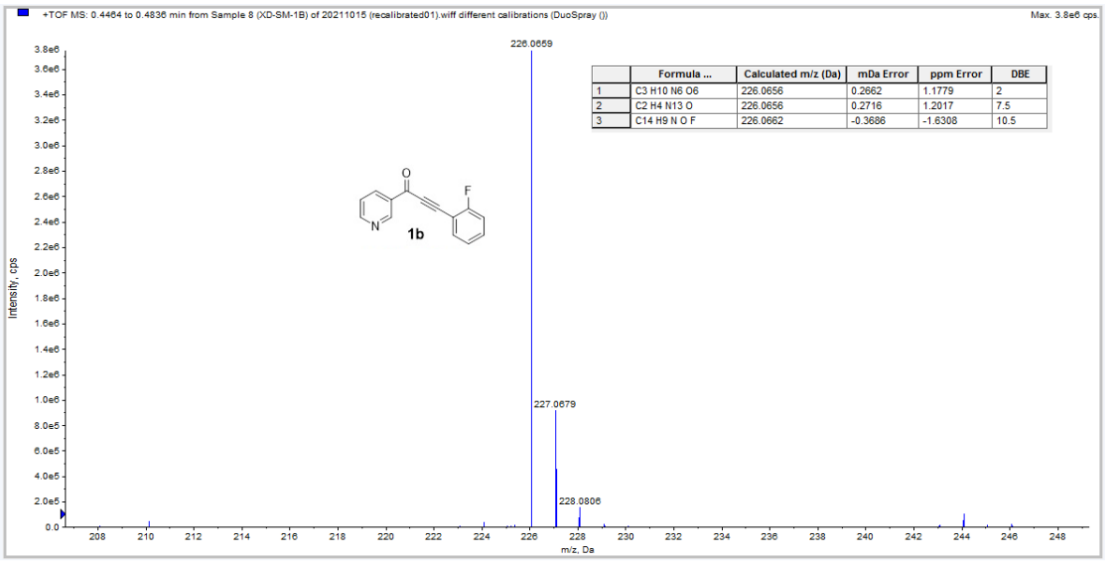
[2] Dermenci, A.; Whittaker, R. E.; Gao, Y.; Cruz, F. A.; Yu, Z.-X.; Dong, G.-B. Rh-catalyzed decarbonylation of conjugated ynones via carbon–alkyne bond activation: reaction scope and mechanistic exploration *via* DFT calculations. *Chem. Sci.* **2015**, *6*, 3201.

[3] Ji, K.-G.; Yang, F.; Gao, S.-Y.; Tang, J.-J.; Gao, J.-M. Gold-Catalyzed Oxidation/CH Functionalization of Ynones: Efficient and Rapid Access to Functionalized Polycyclic Salicyl Ketones. *Chem. Eur. J.* **2016**, *22*, 10225.

[4] Aramini, A.; Brinchi, L.; Germani, R.; Savelli, G. Reductions of α,β-Unsaturated Ketones by $NaBH_4$ or $NaBH_4$ + $CoCl_2$: Selectivity Control by Water or by Aqueous Micellar Solutions. *Eur. J. Org. Chem.* **2000**, 1793.

10. ^{1}H and ^{13}C NMR spectra for substrates and products

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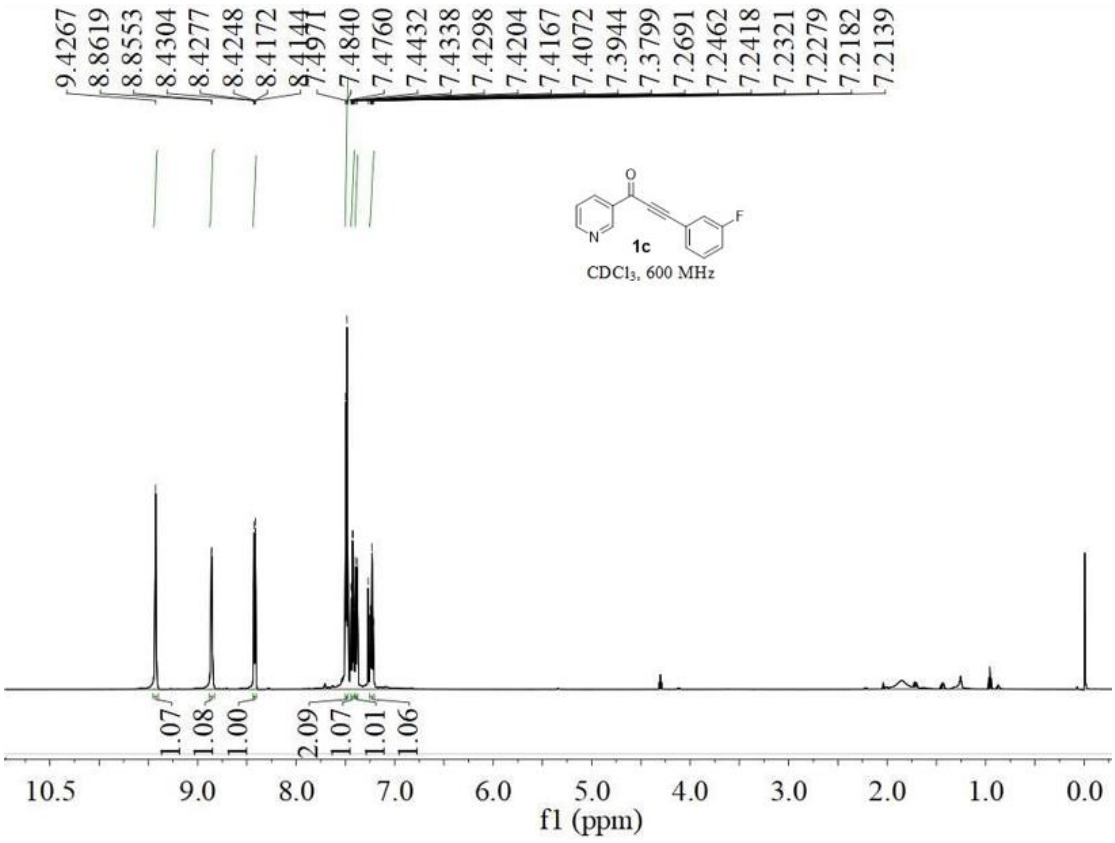
Max. 3.8e6 cps.
Intensity, cps
m/z, Da
226.0659
227.0679
228.0806
1b
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C3 H10 N6 O6 | 226.0656 | 0.2662 | 1.1779 | 2
2 | C2 H4 N13 O | 226.0656 | 0.2716 | 1.2017 | 7.5
3 | C14 H9 N O F | 226.0662 | -0.3686 | -1.6308 | 10.5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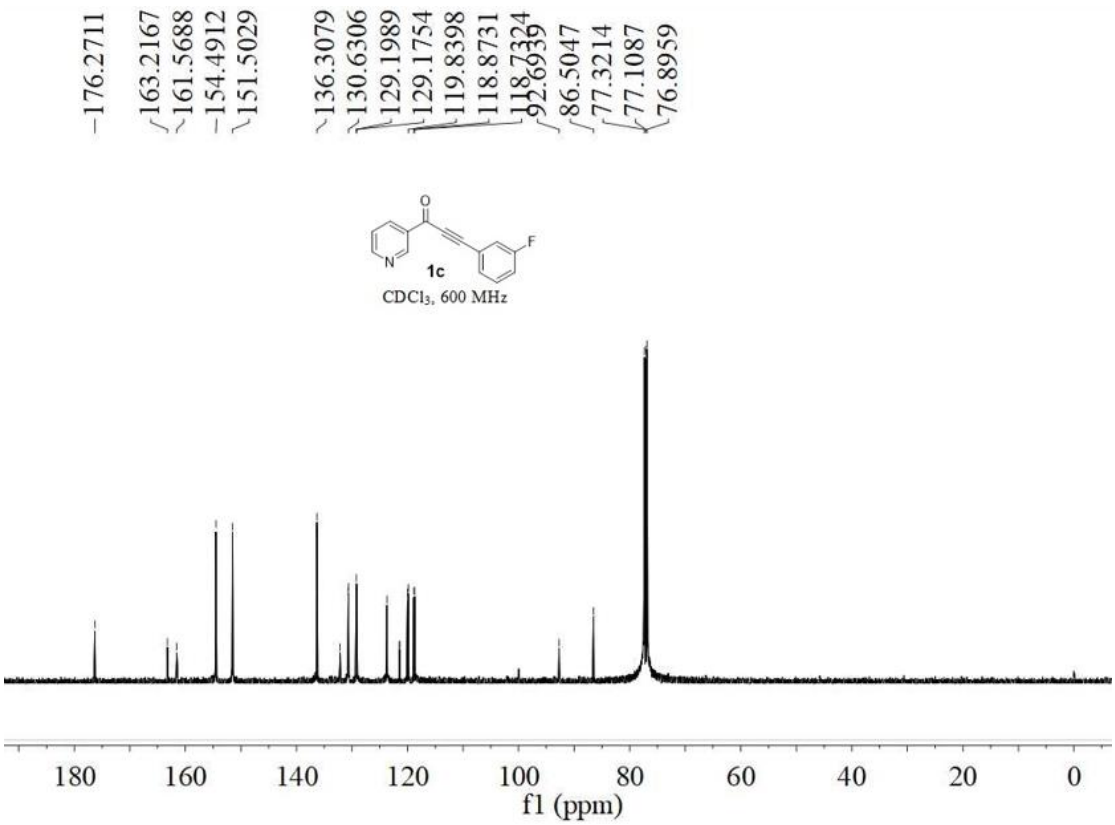
8.8553
8.4304
8.4277
8.4248
8.4172
8.4144
8.4071
7.4840
7.4760
7.4432
7.4338
7.4298
7.4204
7.4167
7.4072
7.3944
7.3799
7.2691
7.2462
7.2418
7.2321
7.2279
7.2182
7.2139
1c
CDCl3, 600 MHz
1.07
1.08
1.00
2.09
1.07
1.01
1.06
f1 (ppm)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161.5688
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129.1754
119.8398
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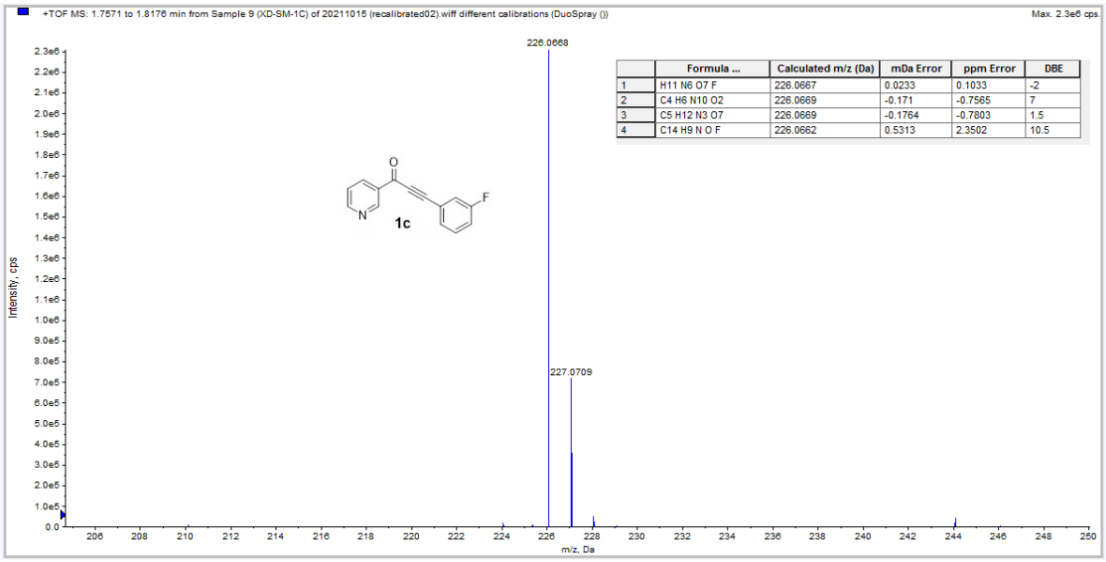
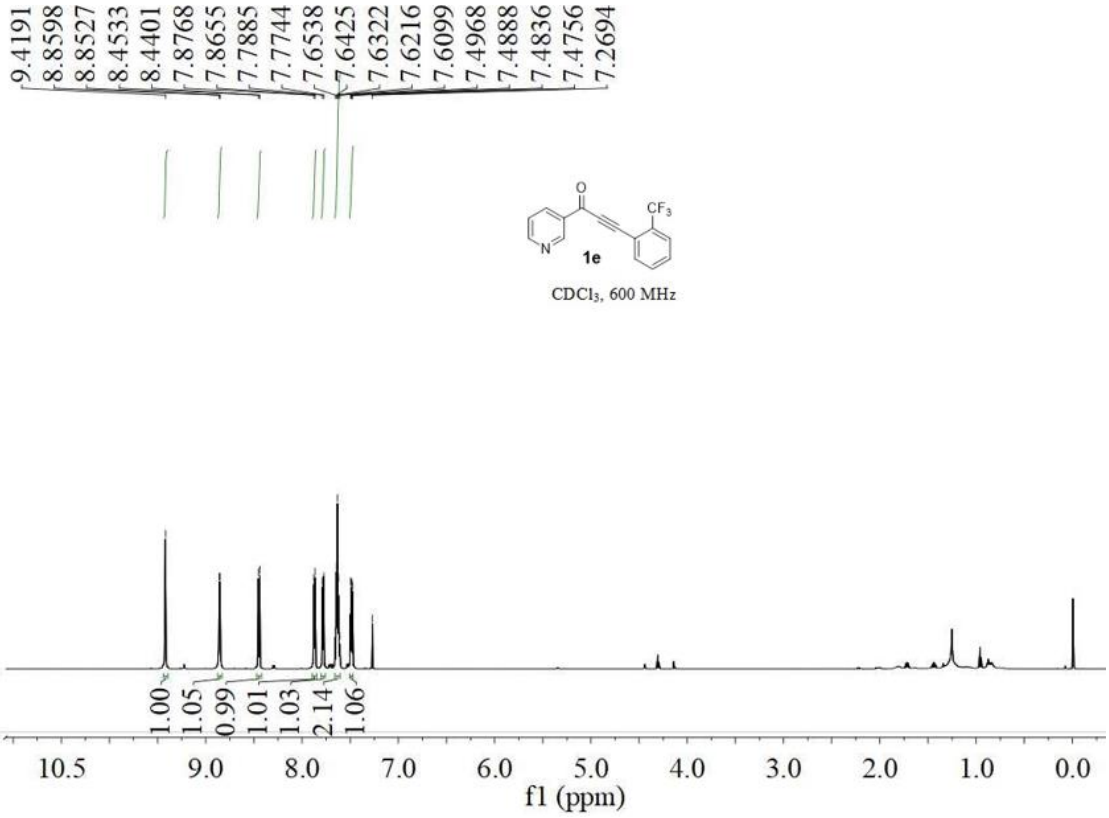
118.7324
92.6939
86.5047
77.3214
77.1087
76.8959
1c
CDCl3, 600 MHz
180
160
140
120
100
80
60
40
20
0
f1 (ppm)


+TOF MS: 1.7571 to 1.8176 min from Sample 9 (XD-SM-1C) of 20211015 (recalibrated02).wiff different calibrations (DuoSpray ())
Max. 2.3e6 cps.
226.0668
227.0709
Intensity, cps
m/z, Da
1c
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | H11 N6 O7 F | 226.0667 | 0.0233 | 0.1033 | -2
2 | C4 H6 N10 O2 | 226.0669 | -0.171 | -0.7565 | 7
3 | C5 H12 N3 O7 | 226.0669 | -0.1764 | -0.7803 | 1.5
4 | C14 H9 N O F | 226.0662 | 0.5313 | 2.3502 | 10.5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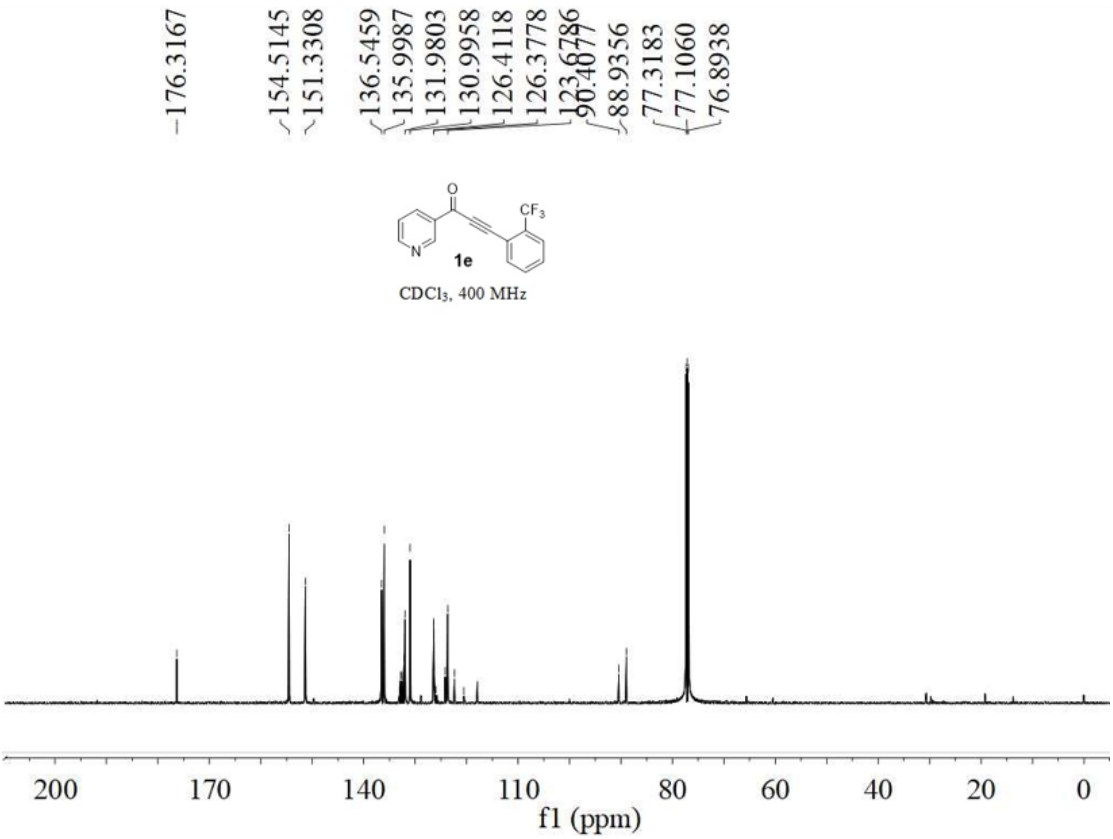
7.4968
7.4888
7.4836
7.4756
7.2694
O
CF3
N
1e
CDCl3, 600 MHz
1.00
1.05
0.99
1.01
1.03
2.14
1.06
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)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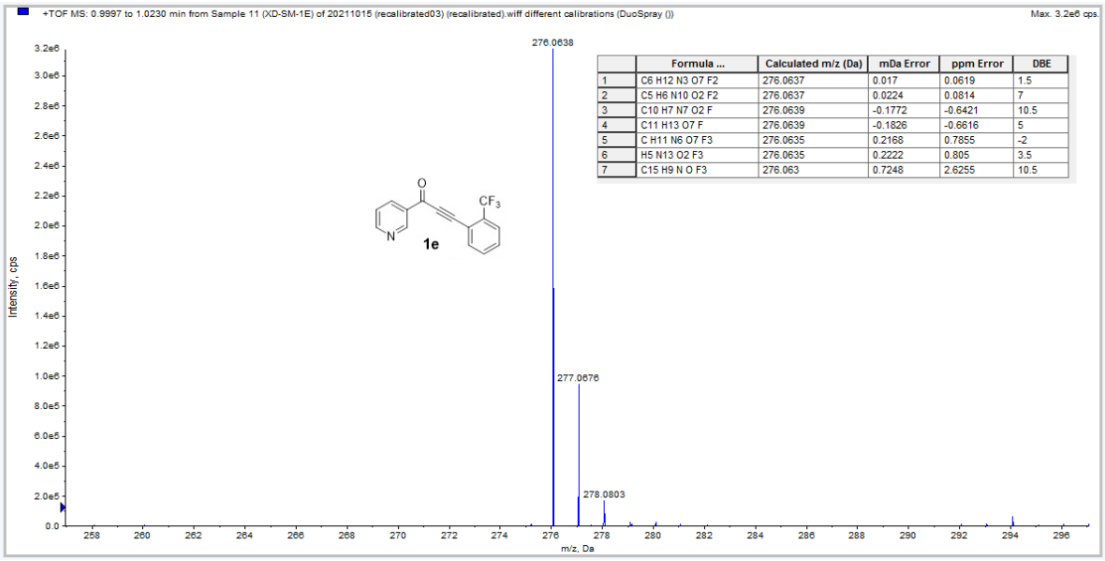
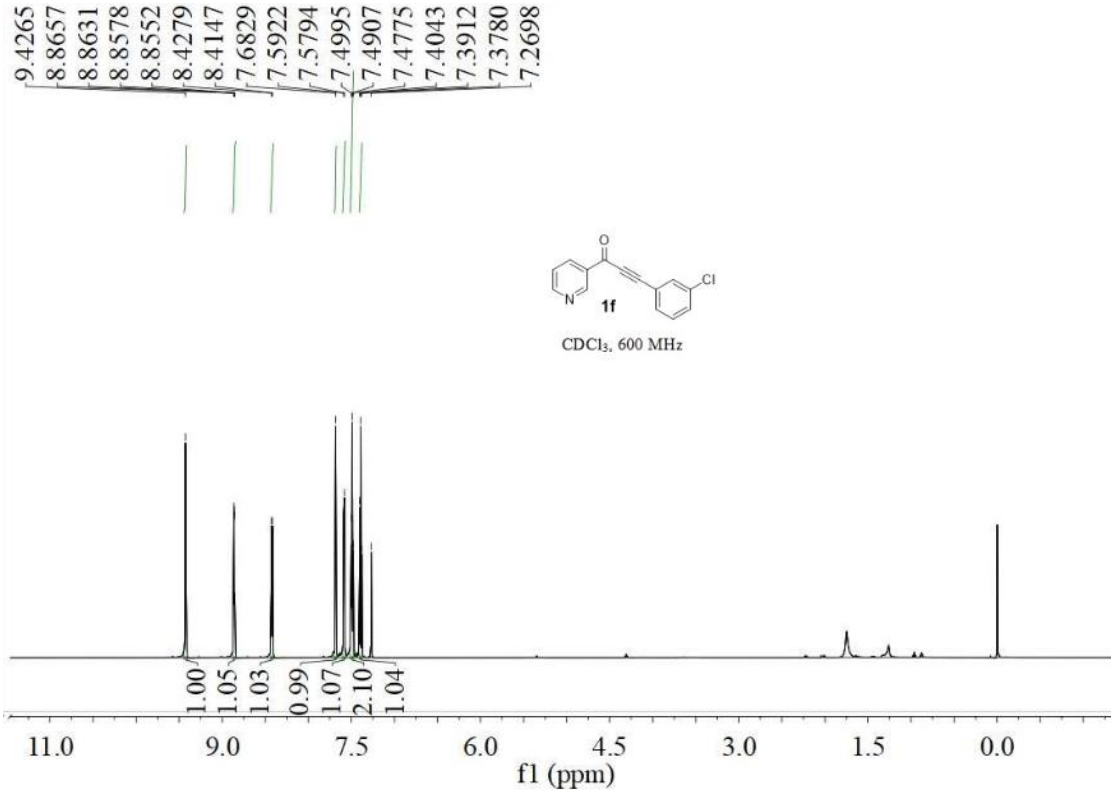
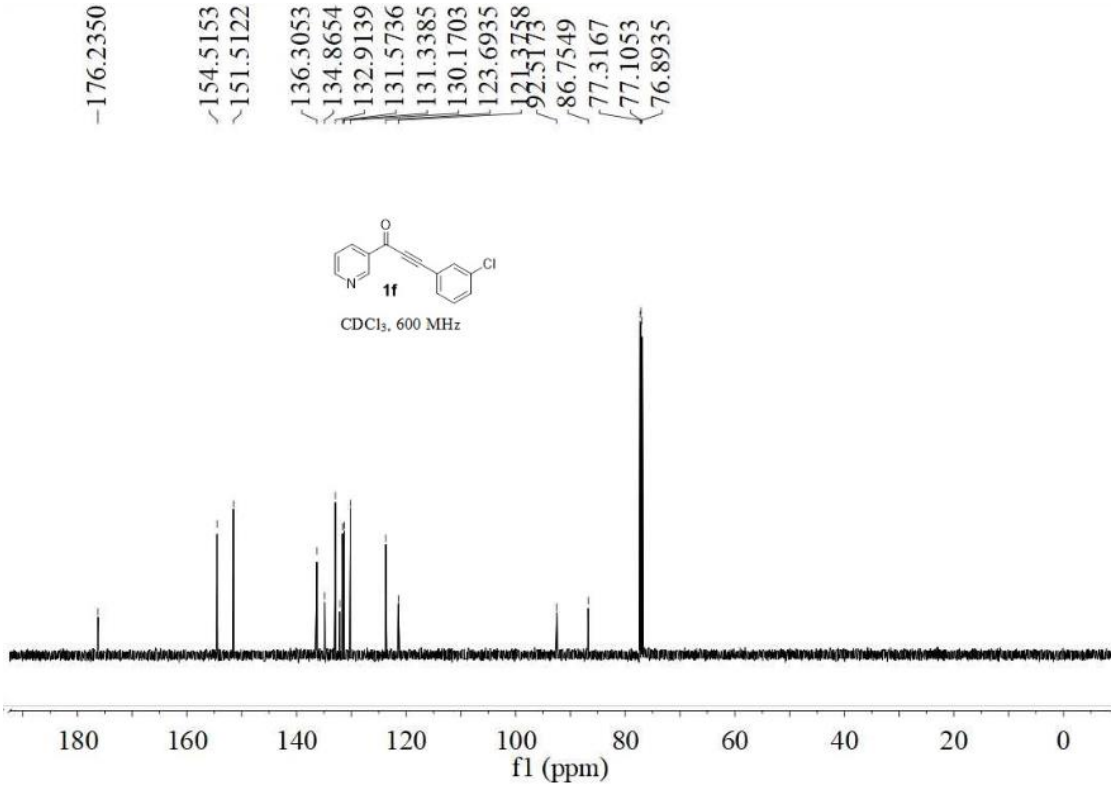
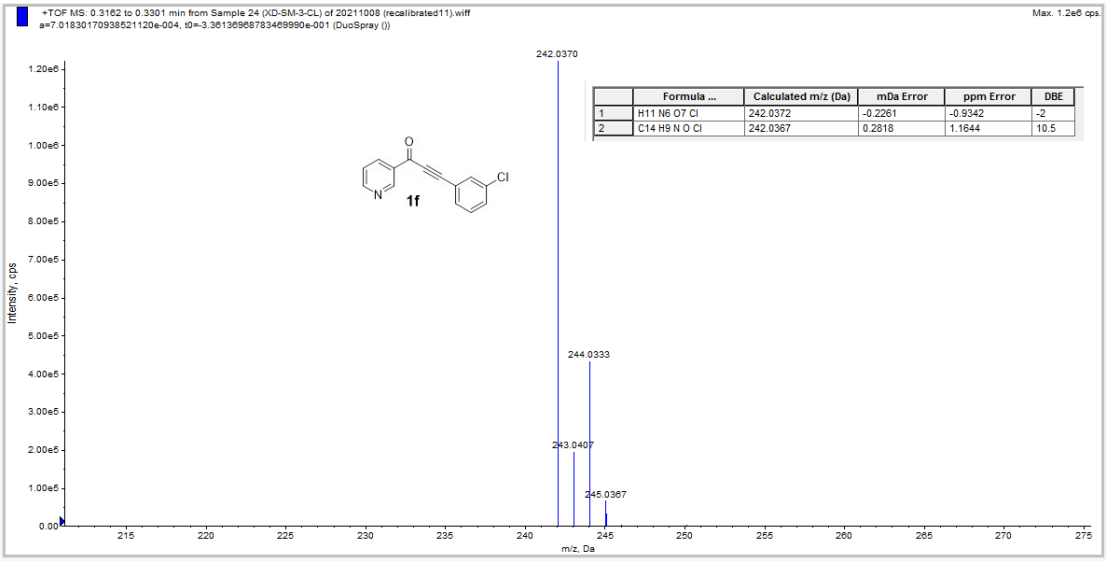
77.3183
77.1060
76.8938
O
CF3
N
1e
CDCl3, 400 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

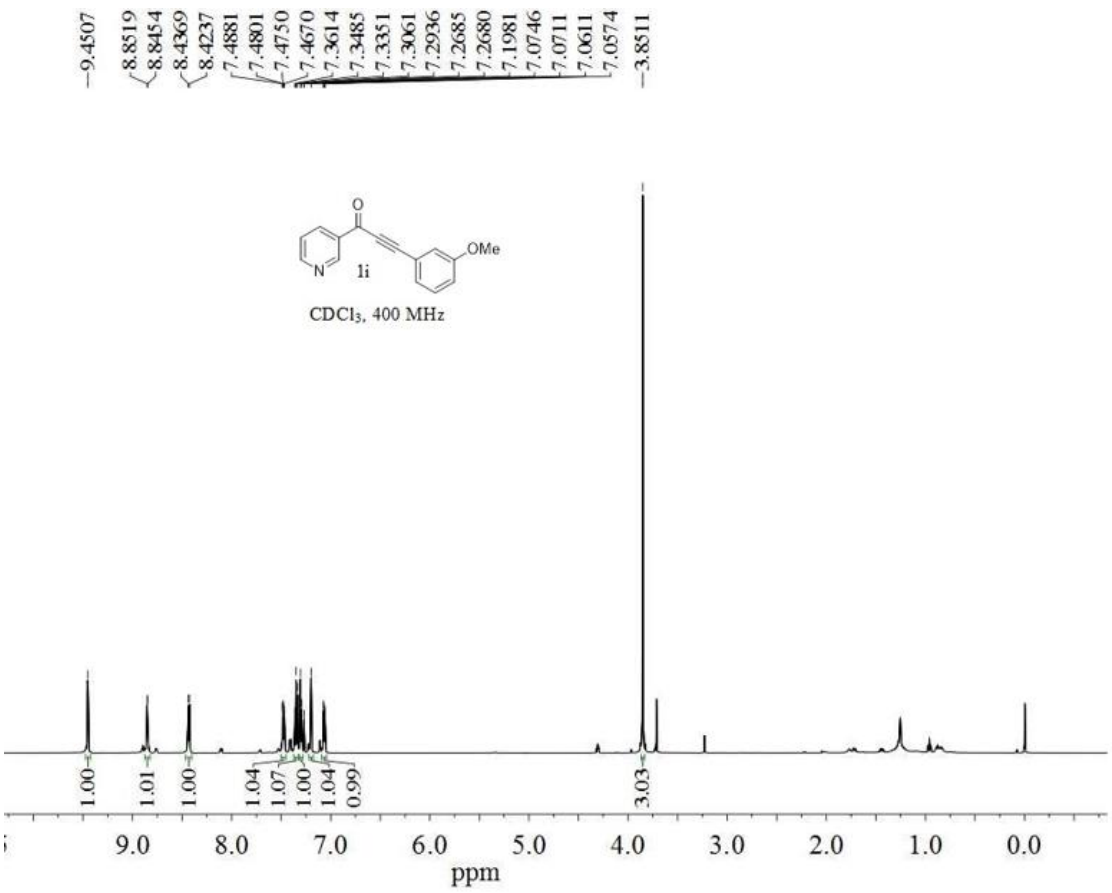


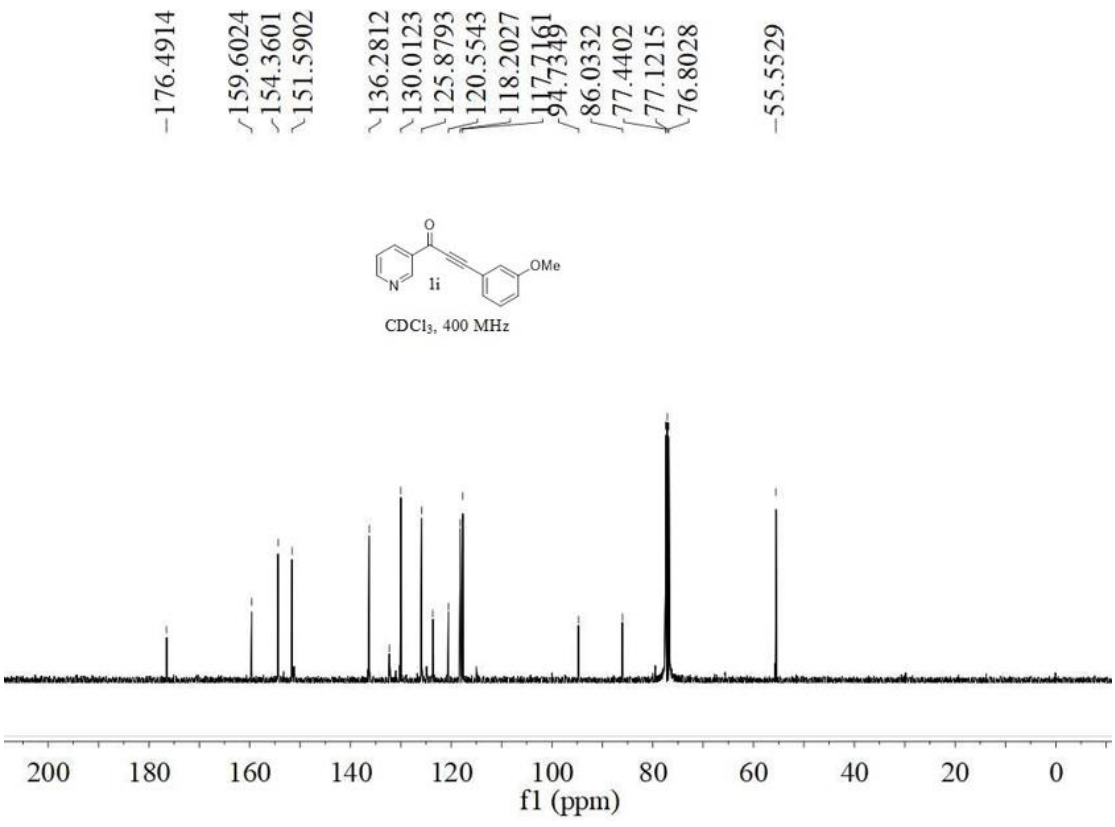
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C6 H12 N3 O7 F2	276.0637	0.017	0.0619	1.5
2	C5 H6 N10 O2 F2	276.0637	0.0224	0.0814	7
3	C10 H7 N7 O2 F	276.0639	-0.1772	-0.6421	10.5
4	C11 H13 O7 F	276.0639	-0.1826	-0.6616	5
5	C H11 N6 O7 F3	276.0635	0.2168	0.7855	-2
6	H5 N13 O2 F3	276.0635	0.2222	0.805	3.5
7	C15 H9 N O F3	276.063	0.7248	2.6255	10.5

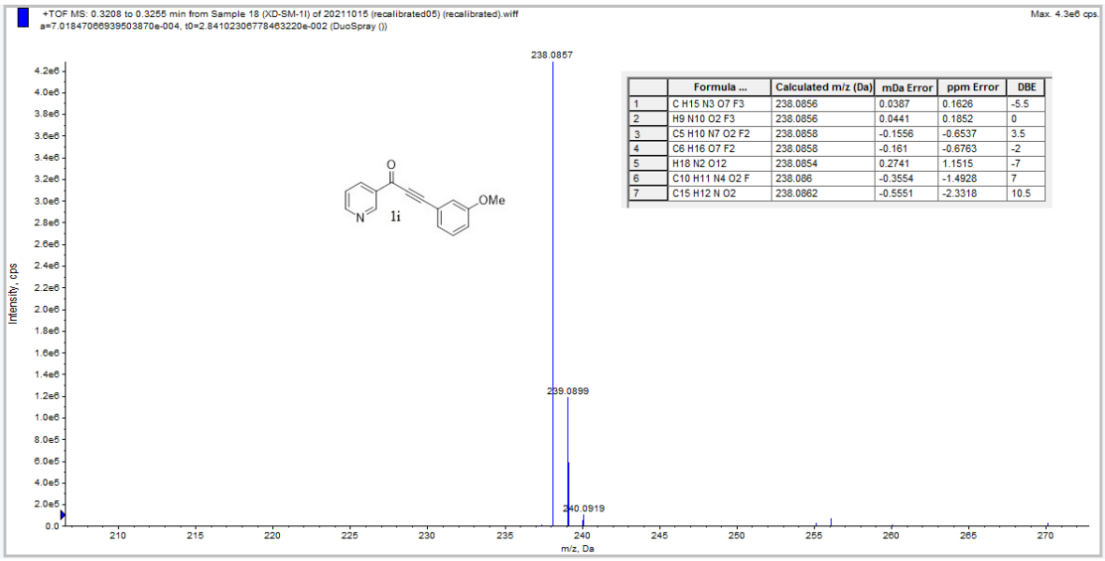


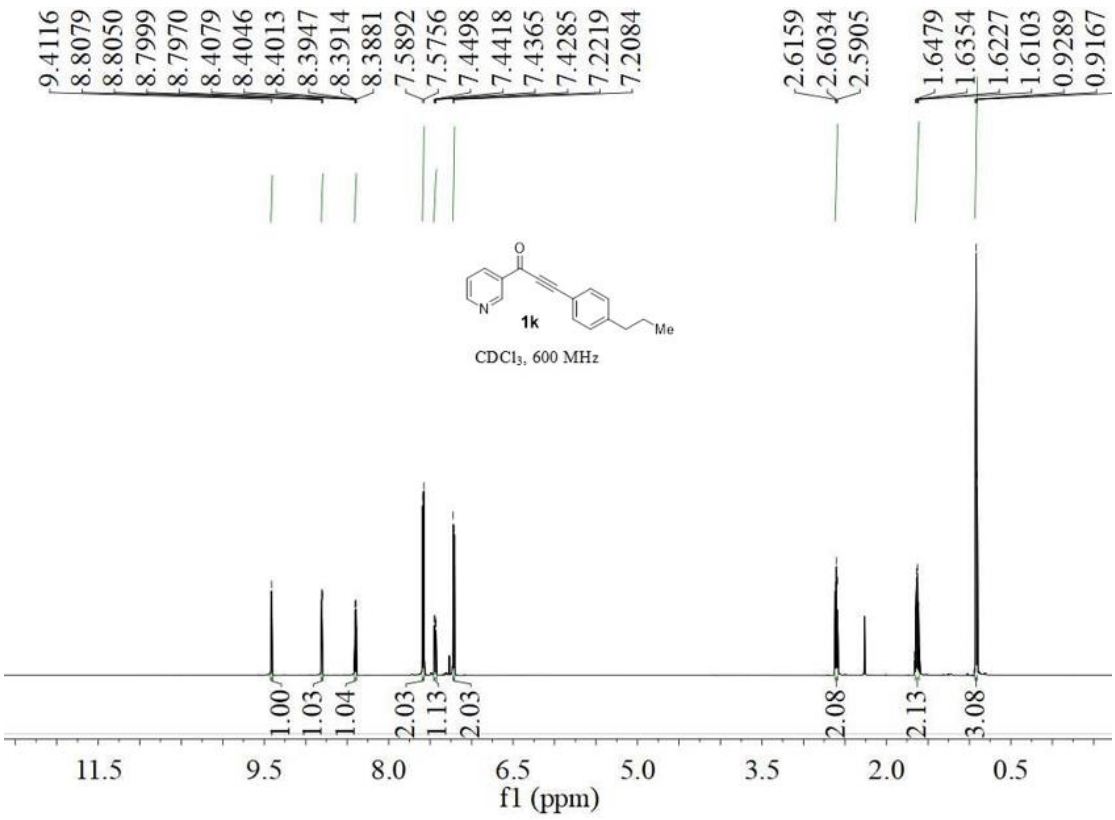

176.2350
154.5153
151.5122
136.3053
134.8654
132.9139
131.5736
131.3385
130.1703
123.6935
121.3758
92.5173
86.7549
77.3167
77.1053
76.8935
O
N
Cl
1f
CDCl3, 600 MHz
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

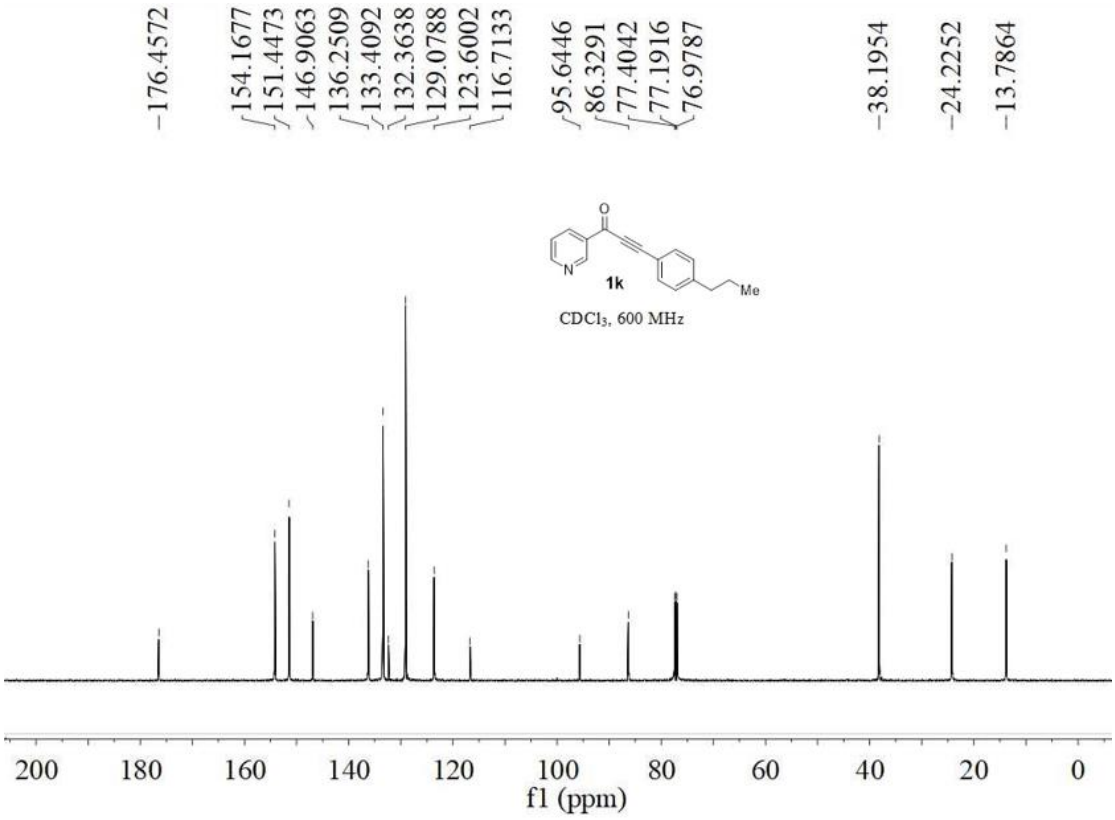

+TOF MS: 0.3162 to 0.3301 min from Sample 24 (XD-SM-3-CL) of 20211008 (recalibrated11).wiff
a=7.0183017093852112 0e-004, t0=-3.36136968783469990e-001 (DuoSpray ())
Max. 1.2e6 cps
242.0370
244.0333
243.0407
245.0367
Intensity, cps
m/z, Da
Formula ...
Calculated m/z (Da)
mDa Error
ppm Error
DBE
1 H11 N6 O7 Cl 242.0372 -0.2261 -0.9342 -2
2 C14 H9 N O Cl 242.0367 0.2818 1.1644 10.5
O
N
Cl
1f

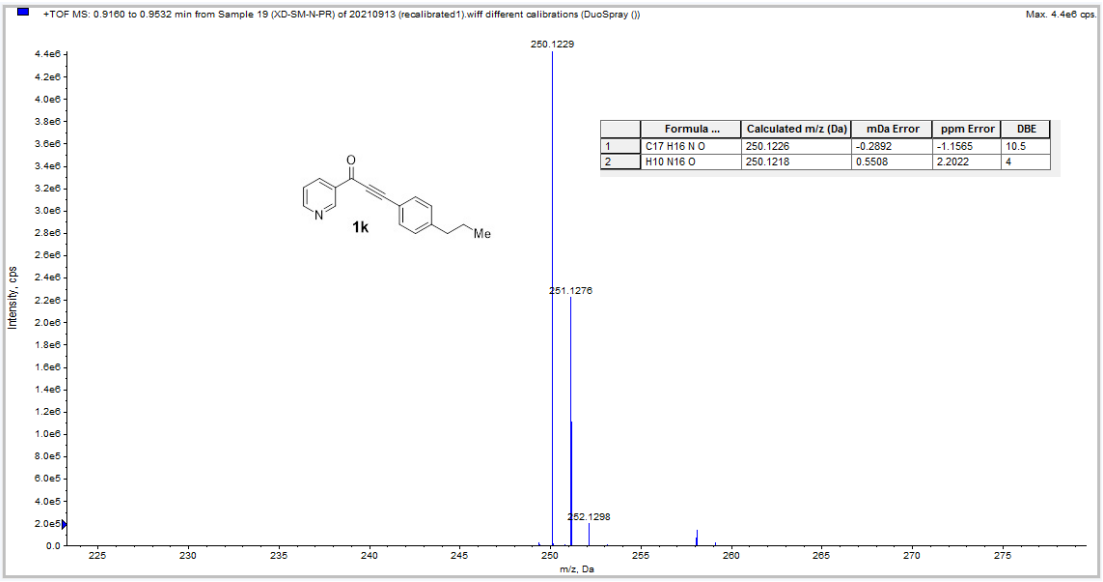

9.4507
8.8519
8.8454
8.4369
8.4237
7.4881
7.4801
7.4750
7.4670
7.3614
7.3485
7.3351
7.3061
7.2936
7.2685
7.2680
7.1981
7.0746
7.0711
7.0611
7.0574
3.8511
O
N
OMe
1i
CDCl3, 400 MHz
1.00
1.01
1.00
1.04
1.07
1.00
1.04
0.99
3.03
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

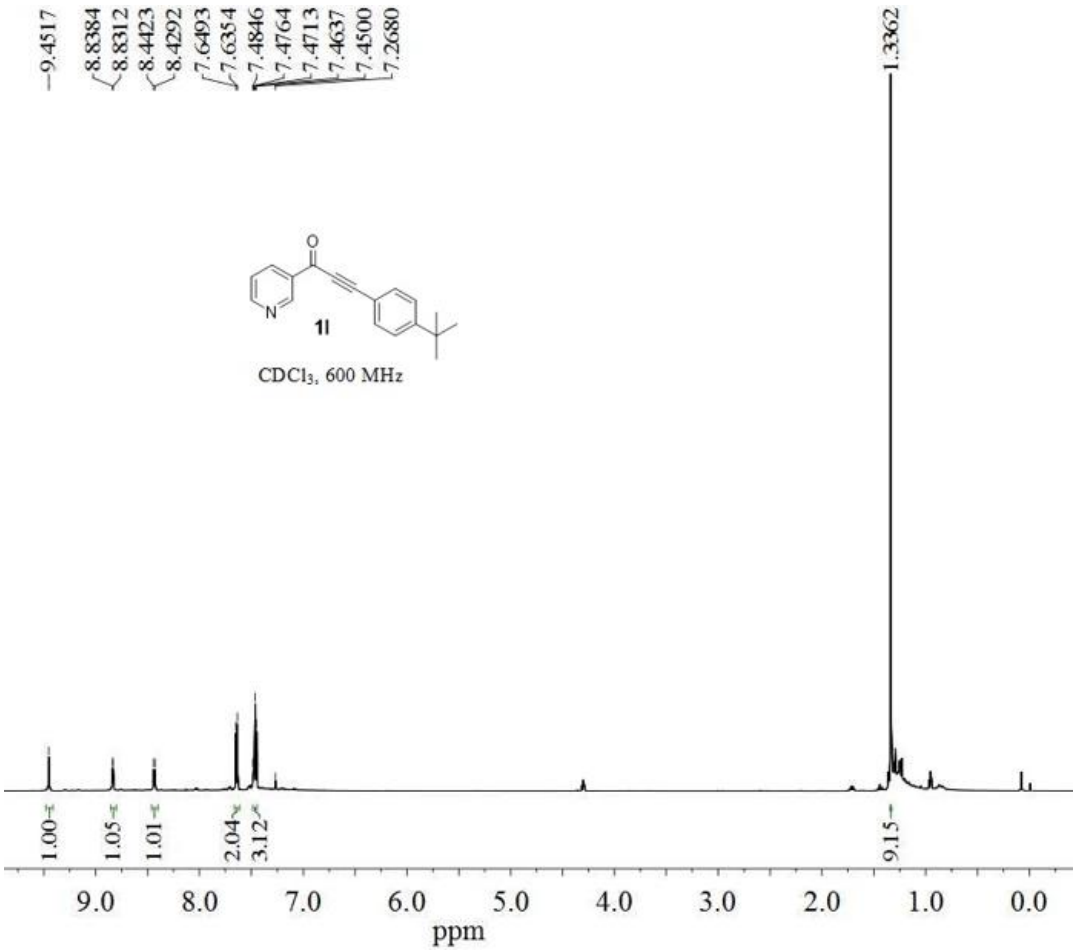

176.4914
159.6024
154.3601
151.5902
136.2812
130.0123
125.8793
120.5543
118.2027
94.7349
86.0332
77.4402
77.1215
76.8028
55.5529
O
N
OMe
1i
CDCl3, 400 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

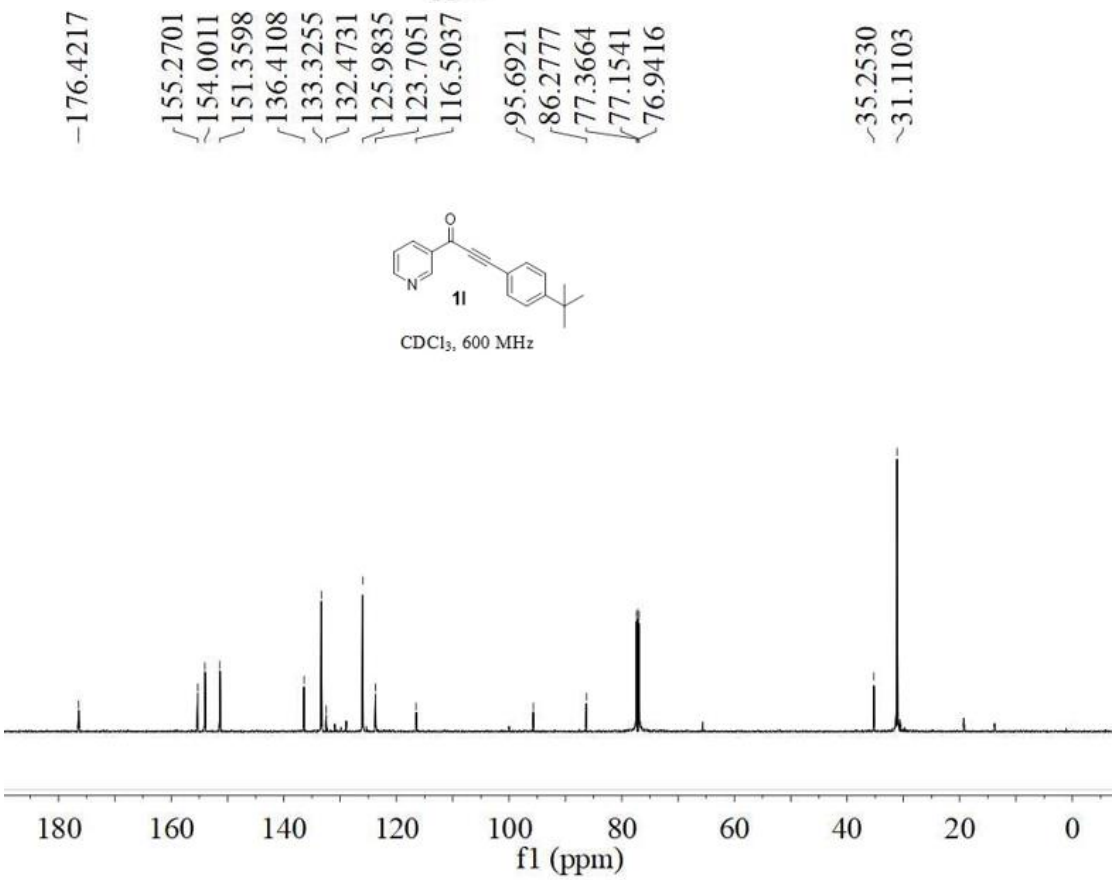

+TOF MS: 0.3208 to 0.3255 min from Sample 18 (XD-SM-1I) of 20211015 (recalibrated05) (recalibrated).wiff
a=7.018470669395038 70e-004, t0=2.841023067784632 20e-002 (DuoSpray ())
Max. 4.3e6 cps
238.0857
239.0899
240.0919
Intensity, cps
m/z, Da
1i
OMe
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C H15 N3 O7 F3 | 238.0856 | 0.0387 | 0.1626 | -5.5
2 | H9 N10 O2 F3 | 238.0856 | 0.0441 | 0.1852 | 0
3 | C5 H10 N7 O2 F2 | 238.0858 | -0.1556 | -0.6537 | 3.5
4 | C6 H16 O7 F2 | 238.0858 | -0.161 | -0.6763 | -2
5 | H18 N2 O12 | 238.0854 | 0.2741 | 1.1515 | -7
6 | C10 H11 N4 O2 F | 238.086 | -0.3554 | -1.4928 | 7
7 | C15 H12 N O2 | 238.0862 | -0.5551 | -2.3318 | 10.5

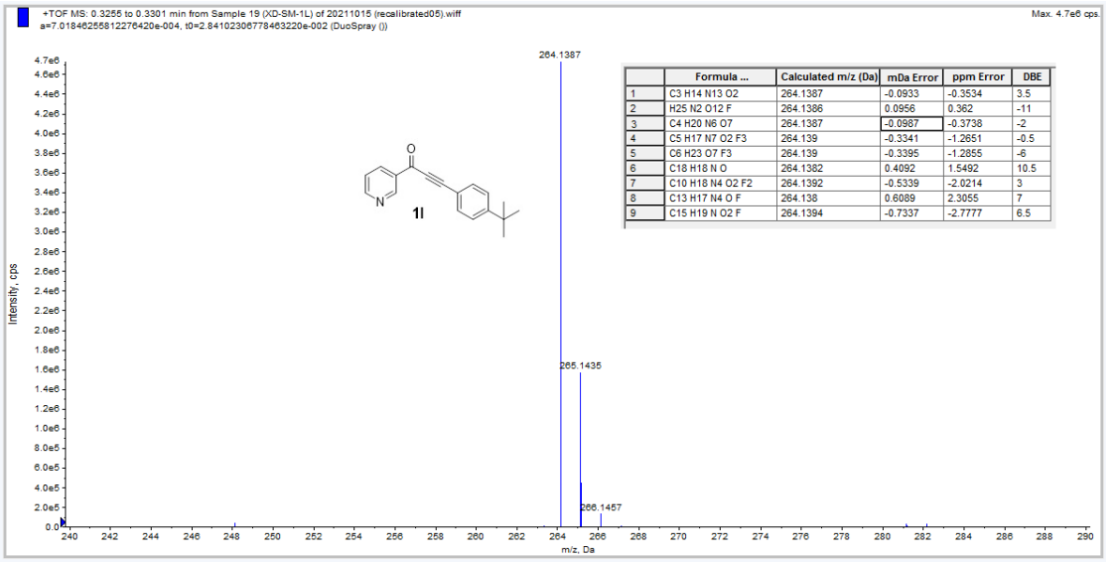

9.4116
8.8079
8.8050
8.7999
8.7970
8.4079
8.4046
8.4013
8.3947
8.3914
8.3881
7.5892
7.5756
7.4498
7.4418
7.4365
7.4285
7.2219
7.2084
2.6159
2.6034
2.5905
1.6479
1.6354
1.6227
1.6103
0.9289
0.9167
1k
Me
CDCl3, 600 MHz
1.00
1.03
1.04
2.03
1.13
2.03
2.08
2.13
3.08
11.5
9.5
8.0
6.5
5.0
3.5
2.0
0.5
f1 (ppm)


176.4572
154.1677
151.4473
146.9063
136.2509
133.4092
132.3638
129.0788
123.6002
116.7133
95.6446
86.3291
77.4042
77.1916
76.9787
38.1954
24.2252
13.7864
1k
Me
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

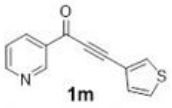

+TOF MS: 0.9160 to 0.9532 min from Sample 19 (XD-SM-N-PR) of 20210913 (recalibrated1).wiff different calibrations (DuoSpray ())
Max. 4.4e6 cps.
250.1229
251.1276
252.1298
1k
Me
Intensity, cps
m/z, Da
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C17 H16 N O | 250.1226 | -0.2892 | -1.1565 | 10.5
2 | H10 N16 O | 250.1218 | 0.5508 | 2.2022 | 4


9.4517
8.8384
8.8312
8.4423
8.4292
7.6493
7.6354
7.4846
7.4764
7.4713
7.4637
7.4500
7.2680
1.3362
1l
CDCl3, 600 MHz
1.00
1.05
1.01
2.04
3.12
9.15
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

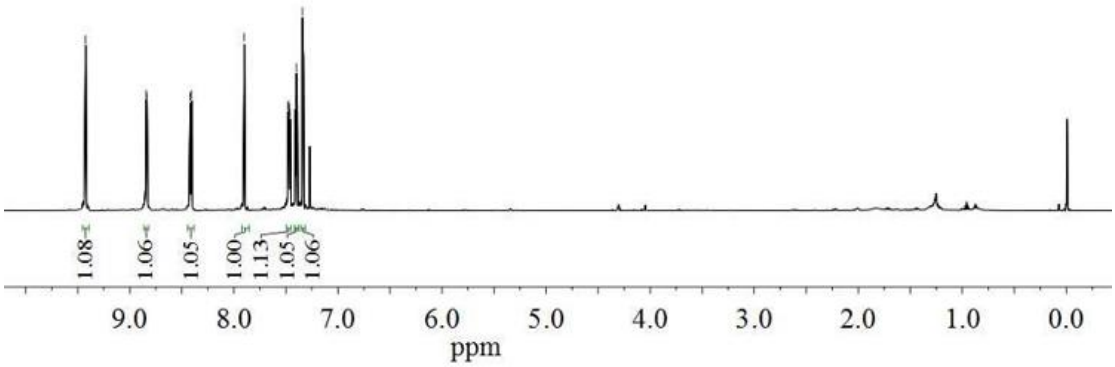

176.4217
155.2701
154.0011
151.3598
136.4108
133.3255
132.4731
125.9835
123.7051
116.5037
95.6921
86.2777
77.3664
77.1541
76.9416
35.2530
31.1103
1l
CDCl3, 600 MHz
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

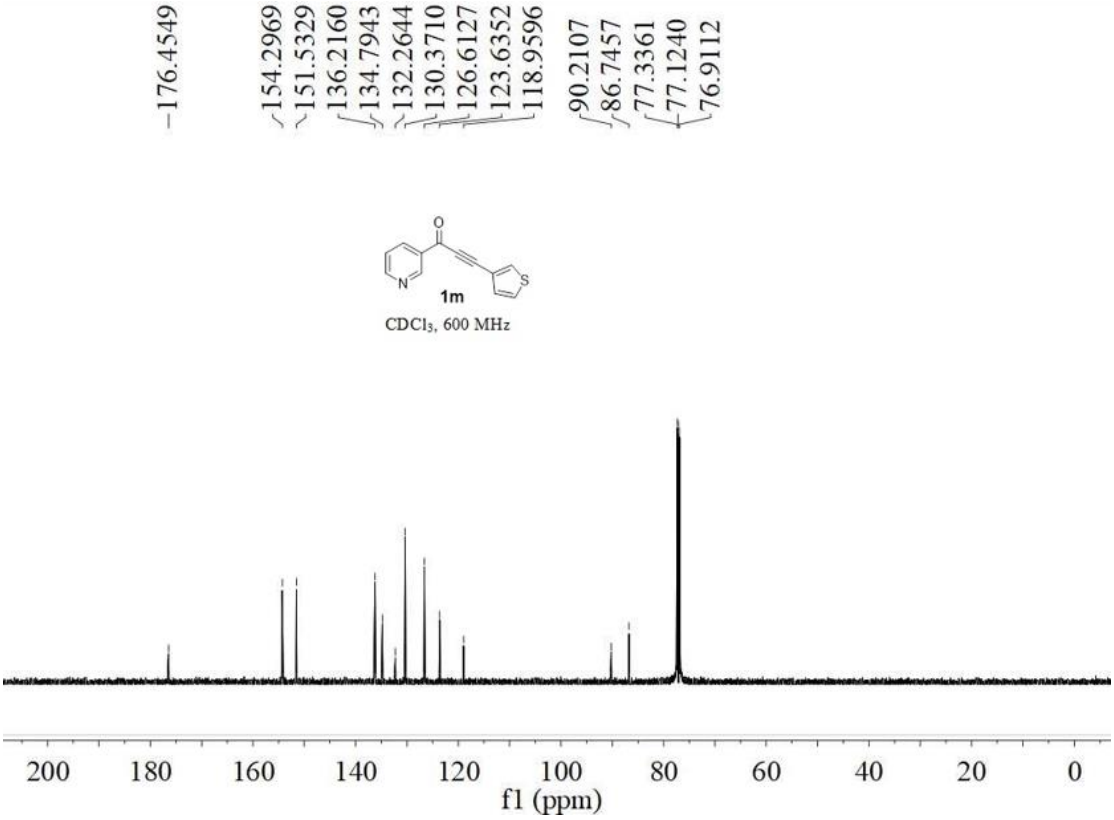


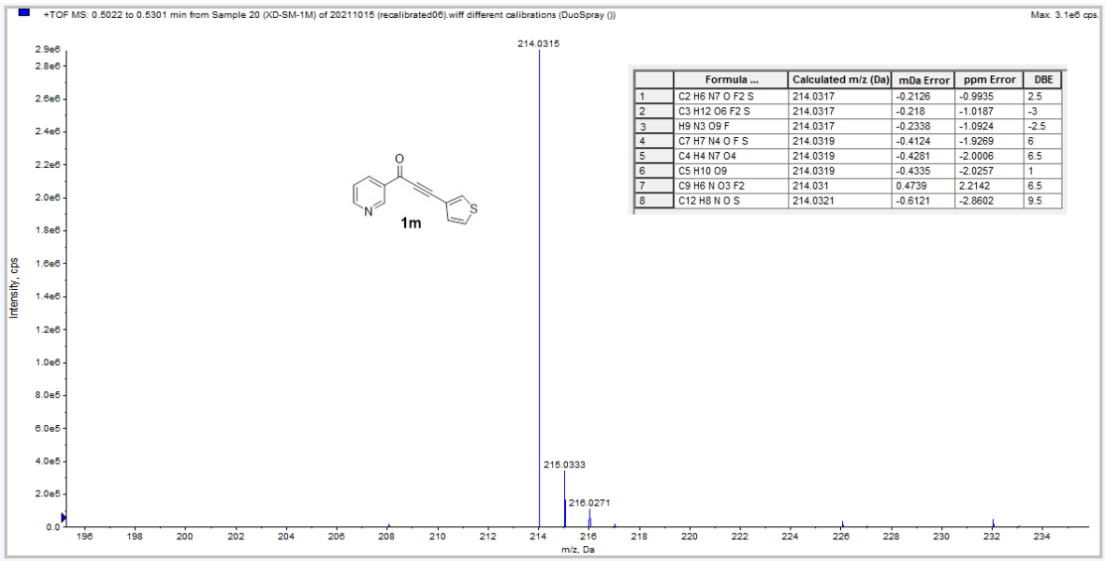
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C3 H14 N13 O2	264.1387	-0.0933	-0.3534	3.5
2	H25 N2 O12 F	264.1386	0.0956	0.362	-11
3	C4 H20 N6 O7	264.1387	-0.0987	-0.3738	-2
4	C5 H17 N7 O2 F3	264.139	-0.3341	-1.2651	-0.5
5	C6 H23 O7 F3	264.139	-0.3395	-1.2855	-6
6	C18 H18 N O	264.1382	0.4092	1.5492	10.5
7	C10 H18 N4 O2 F2	264.1392	-0.5339	-2.0214	3
8	C13 H17 N4 O F	264.138	0.6089	2.3055	7
9	C15 H19 N O2 F	264.1394	-0.7337	-2.7777	6.5

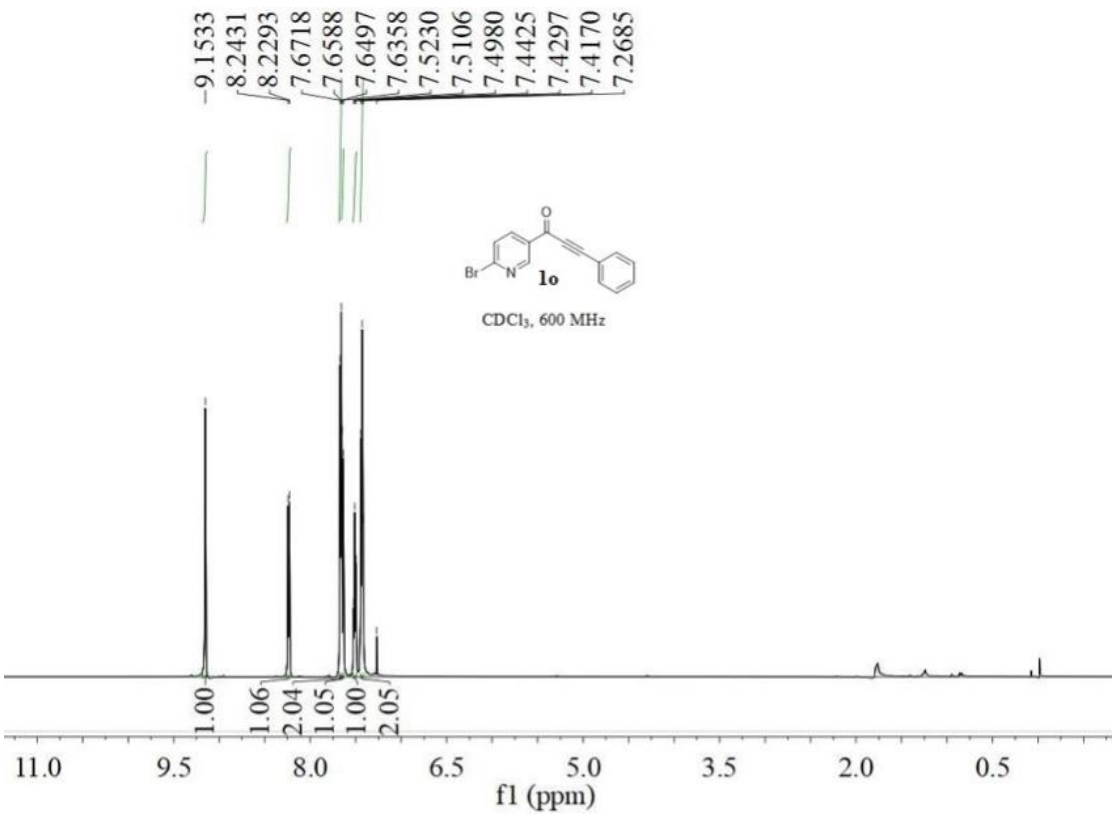


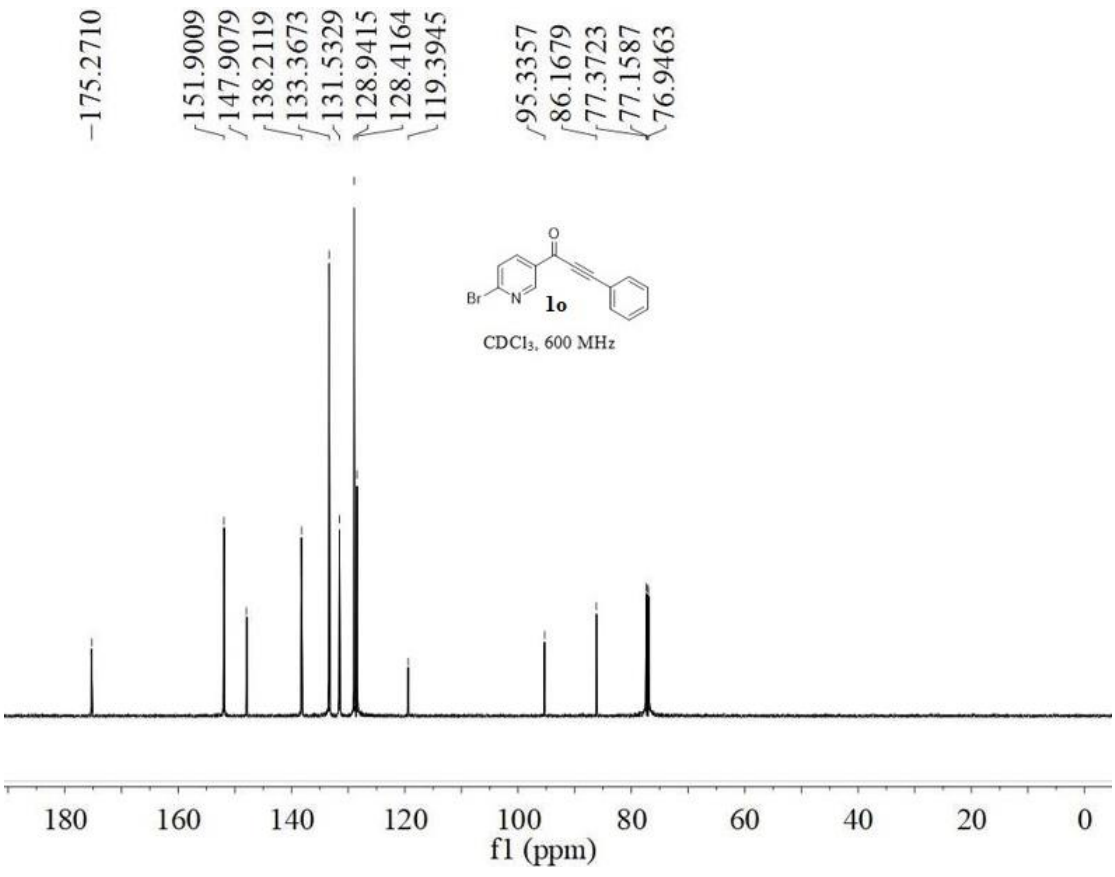
$CDCl_3$, 600 MHz

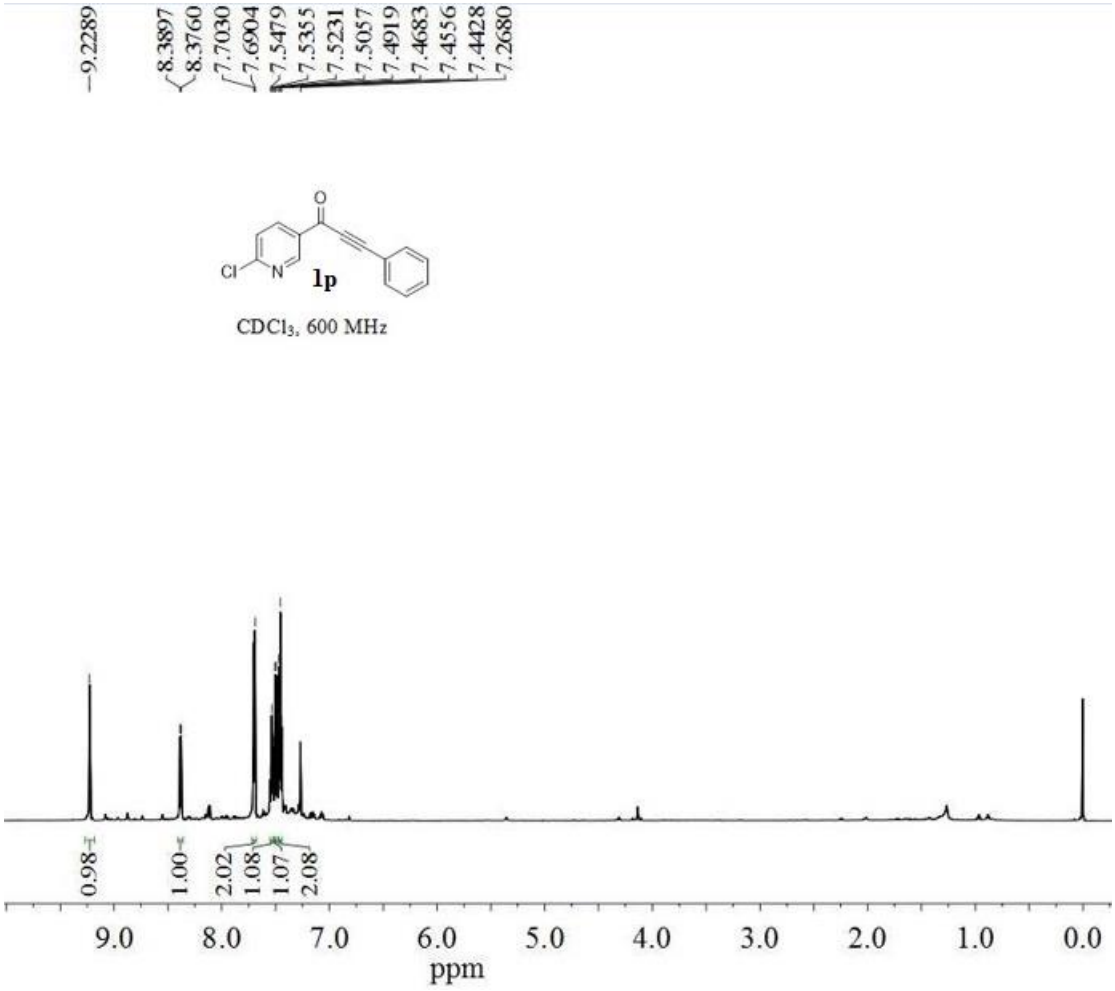


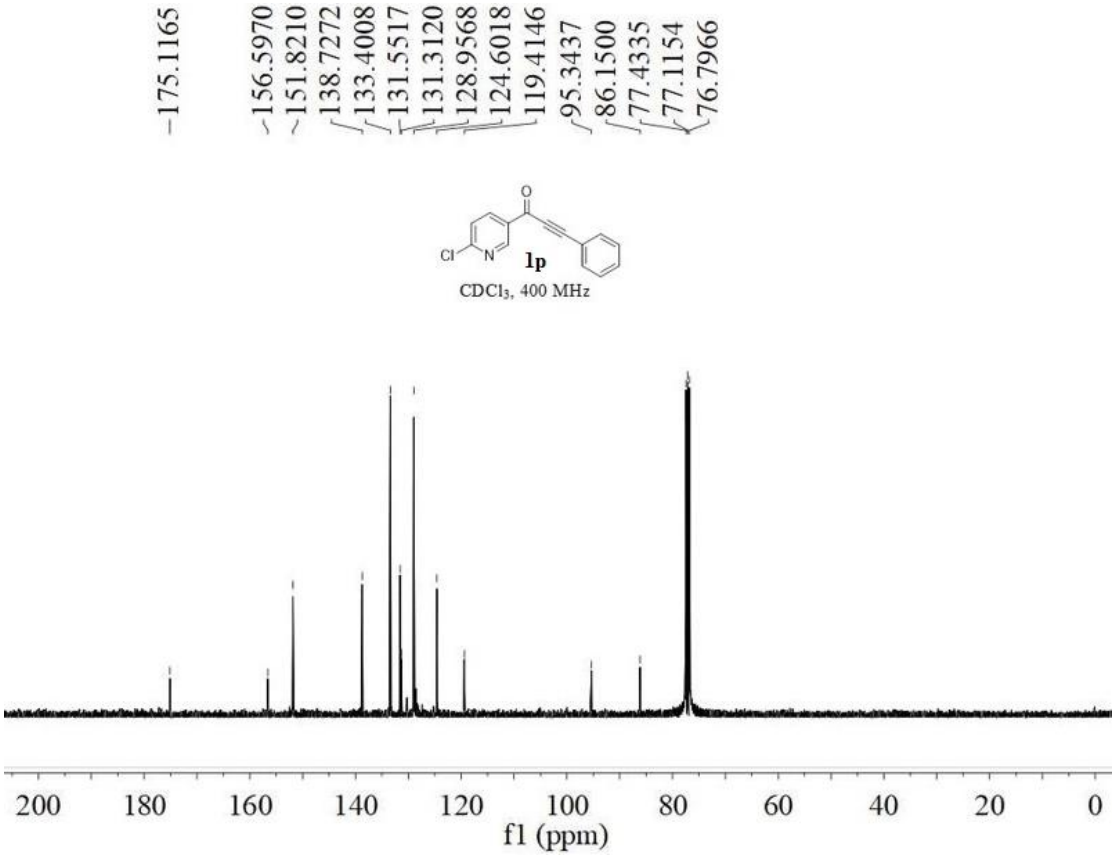

176.4549
154.2969
151.5329
136.2160
134.7943
132.2644
130.3710
126.6127
123.6352
118.9596
90.2107
86.7457
77.3361
77.1240
76.9112
1m
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

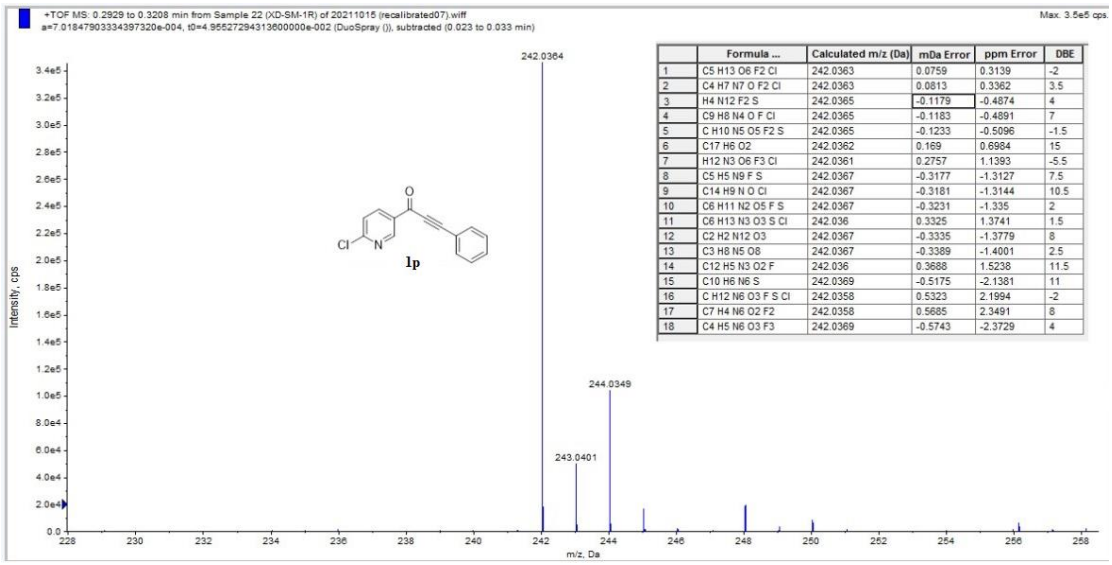

+TOF MS: 0.5022 to 0.5301 min from Sample 20 (XD-SM-1M) of 20211015 (recalibrated06).wiff different calibrations (DuoSpray ())
Max. 3.1e6 cps.
214.0315
215.0333
216.0271
Intensity, cps
m/z, Da
1m
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C2 H6 N7 O F2 S | 214.0317 | -0.2126 | -0.9935 | 2.5
2 | C3 H12 O6 F2 S | 214.0317 | -0.218 | -1.0187 | -3
3 | H9 N3 O9 F | 214.0317 | -0.2338 | -1.0924 | -2.5
4 | C7 H7 N4 O F S | 214.0319 | -0.4124 | -1.9269 | 6
5 | C4 H4 N7 O4 | 214.0319 | -0.4281 | -2.0006 | 6.5
6 | C5 H10 O9 | 214.0319 | -0.4335 | -2.0257 | 1
7 | C9 H6 N O3 F2 | 214.031 | 0.4739 | 2.2142 | 6.5
8 | C12 H8 N O S | 214.0321 | -0.6121 | -2.8602 | 9.5


9.1533
8.2431
8.2293
7.6718
7.6588
7.6497
7.6358
7.5230
7.5106
7.4980
7.4425
7.4297
7.4170
7.2685
Br
N
O
1o
CDCl3, 600 MHz
1.00
1.06
2.04
1.05
1.00
2.05
11.0
9.5
8.0
6.5
5.0
3.5
2.0
0.5
f1 (ppm)

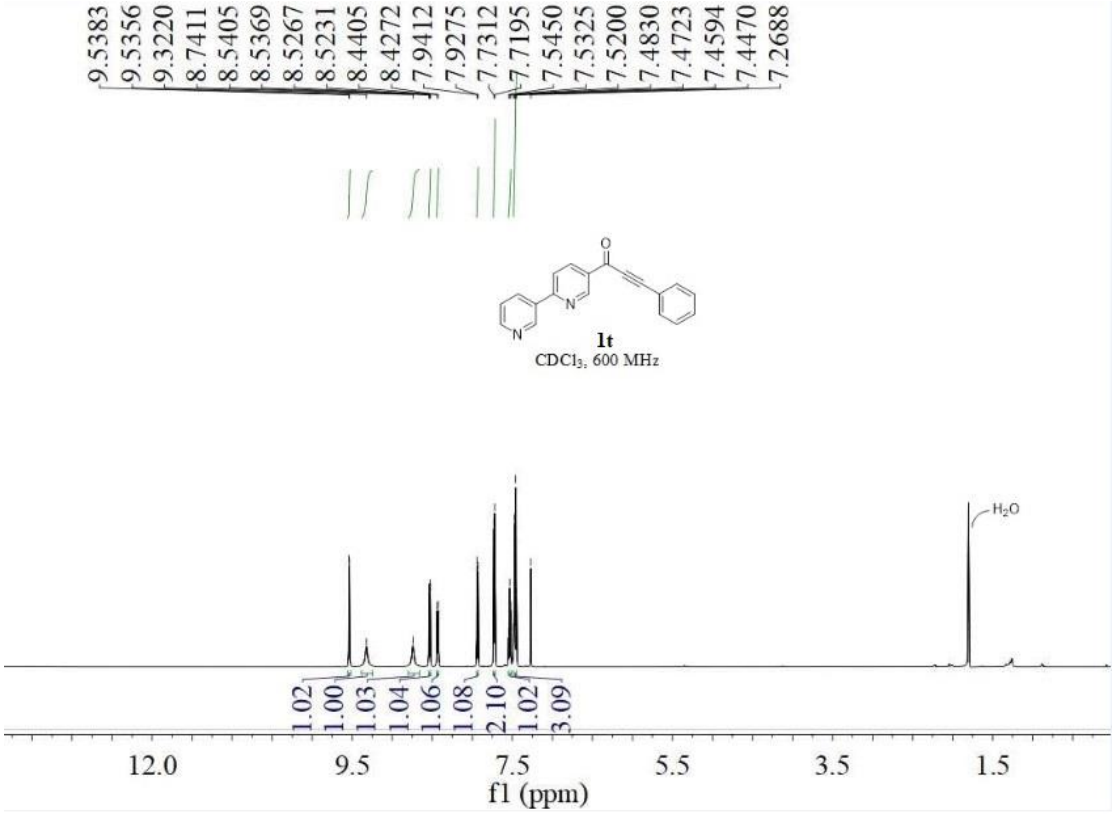

175.2710
151.9009
147.9079
138.2119
133.3673
131.5329
128.9415
128.4164
119.3945
95.3357
86.1679
77.3723
77.1587
76.9463
Br
N
O
1o
CDCl3, 600 MHz
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

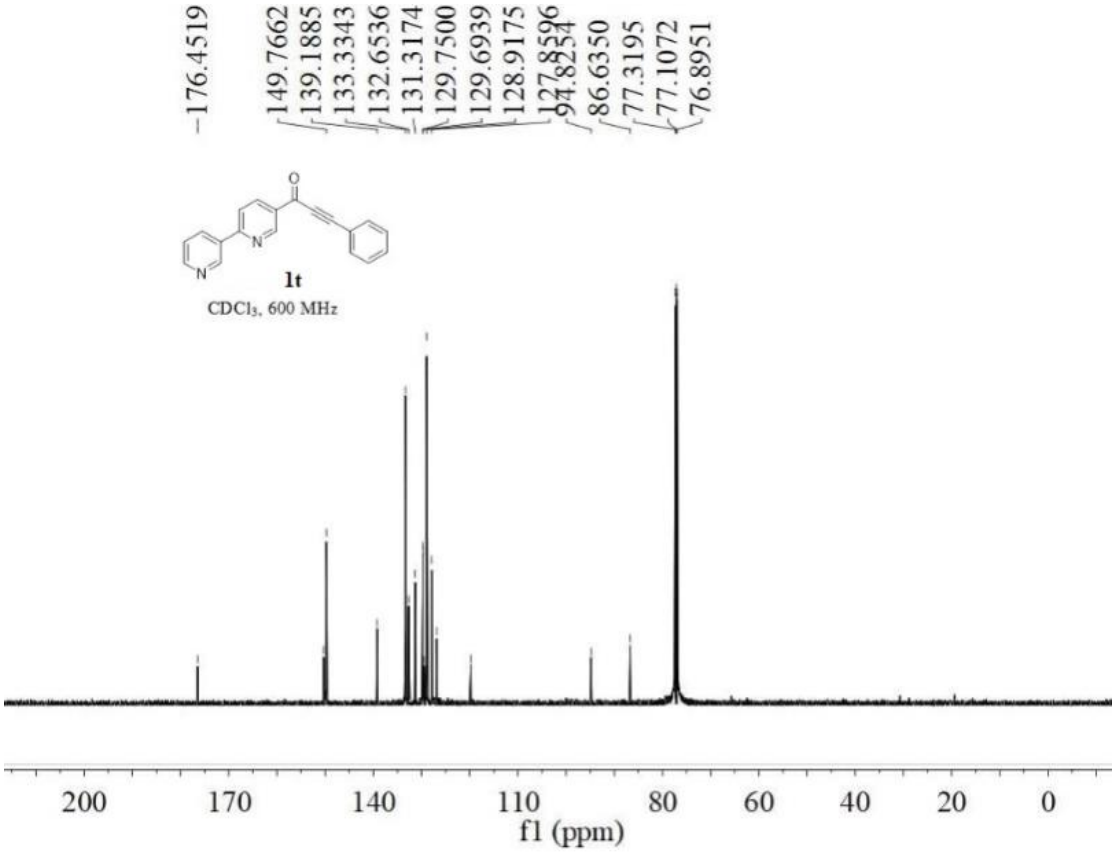

9.2289
8.3897
8.3760
7.7030
7.6904
7.5479
7.5355
7.5231
7.5057
7.4919
7.4683
7.4556
7.4428
7.2680
Cl
N
O
1p
CDCl3, 600 MHz
0.98
1.00
2.02
1.08
1.07
2.08
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
ppm

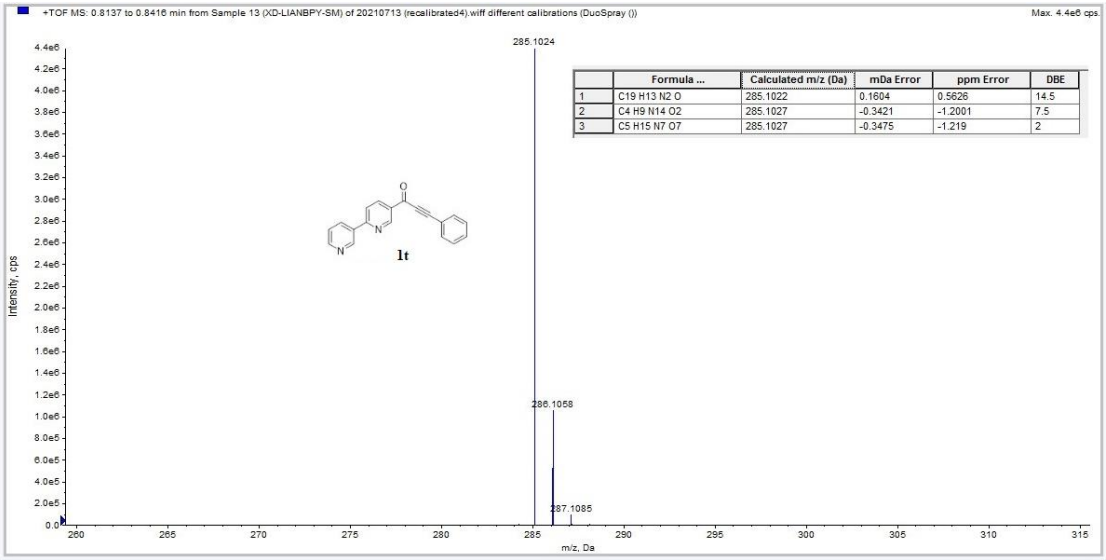

175.1165
156.5970
151.8210
138.7272
133.4008
131.5517
131.3120
128.9568
124.6018
119.4146
95.3437
86.1500
77.4335
77.1154
76.7966
Cl
N
O
1p
CDCl3, 400 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

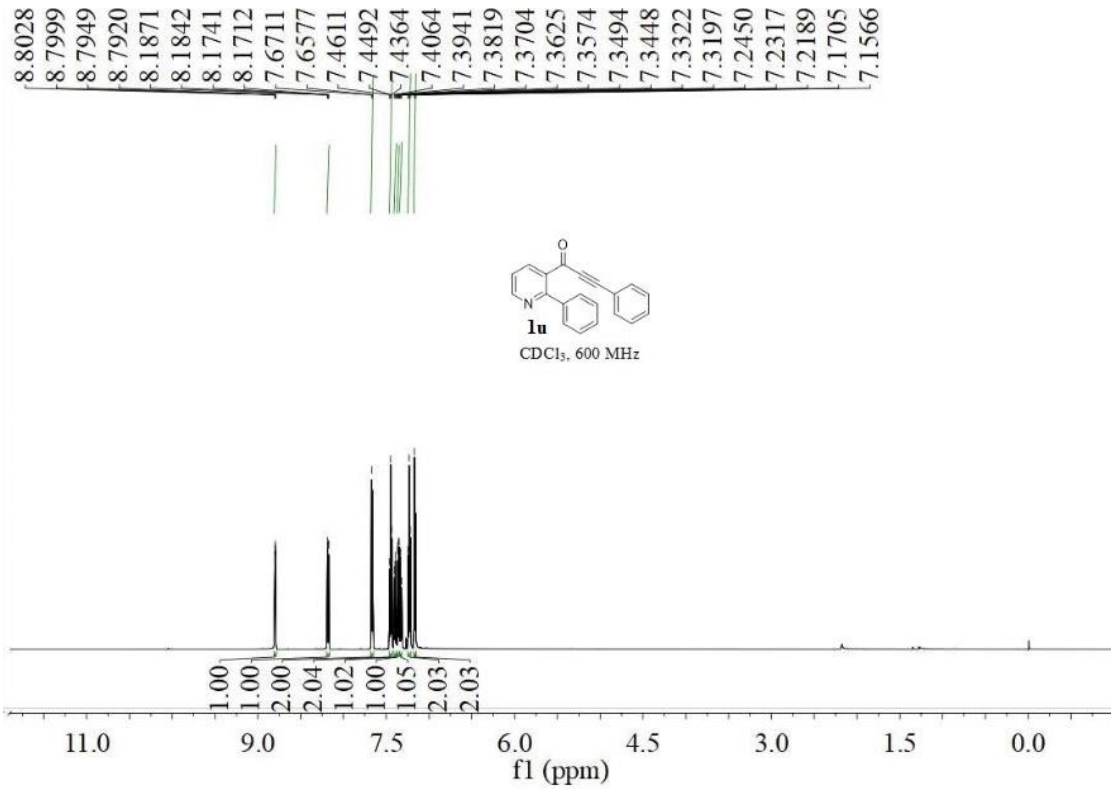


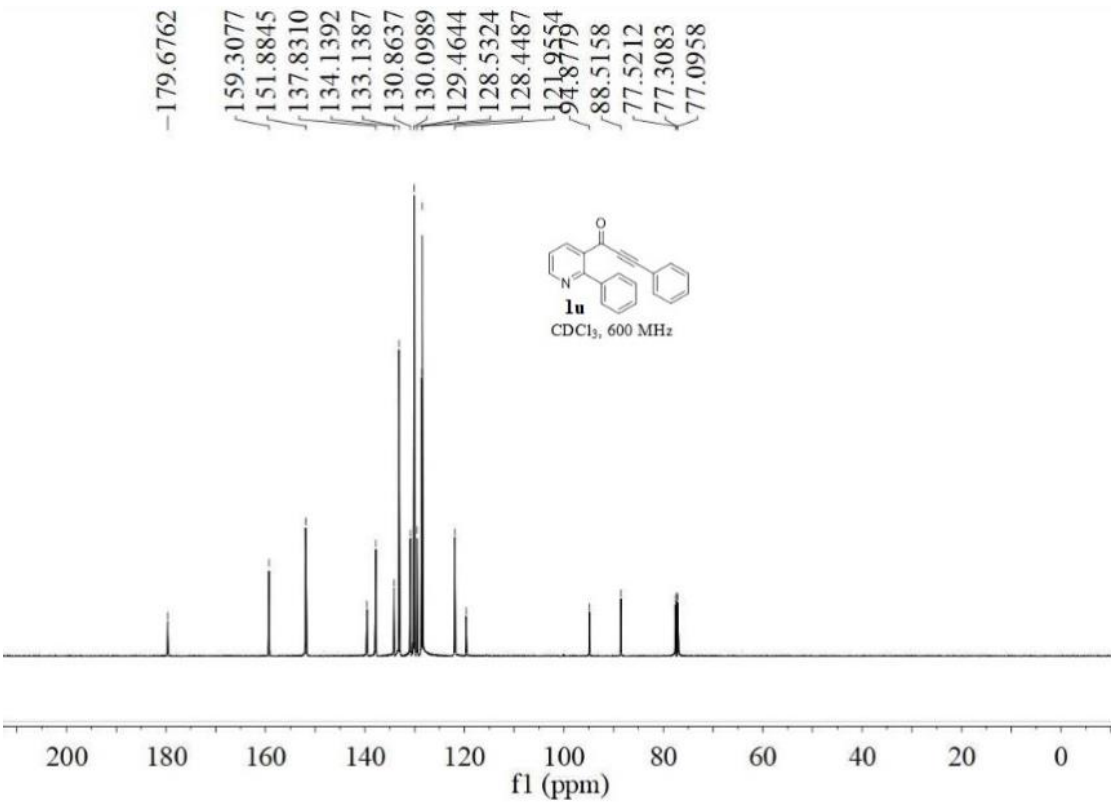
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C5 H13 O6 F2 Cl	242.0363	0.0759	0.3139	-2
2	C4 H7 N7 O F2 Cl	242.0363	0.0813	0.3362	3.5
3	H4 N12 F2 S	242.0365	-0.1179	-0.4874	4
4	C9 H8 N4 O F Cl	242.0365	-0.1183	-0.4891	7
5	C H10 N5 O5 F2 S	242.0365	-0.1233	-0.5096	-1.5
6	C17 H6 O2	242.0362	0.169	0.6984	15
7	H12 N3 O6 F3 Cl	242.0361	0.2757	1.1393	-5.5
8	C5 H5 N9 F S	242.0367	-0.3177	-1.3127	7.5
9	C14 H9 N O Cl	242.0367	-0.3181	-1.3144	10.5
10	C6 H11 N2 O5 F S	242.0367	-0.3231	-1.335	2
11	C6 H13 N3 O3 S Cl	242.036	0.3325	1.3741	1.5
12	C2 H2 N12 O3	242.0367	-0.3335	-1.3779	8
13	C3 H8 N5 O8	242.0367	-0.3389	-1.4001	2.5
14	C12 H5 N3 O2 F	242.036	0.3688	1.5238	11.5
15	C10 H6 N6 S	242.0369	-0.5175	-2.1381	11
16	C H12 N6 O3 F S Cl	242.0358	0.5323	2.1994	-2
17	C7 H4 N6 O2 F2	242.0358	0.5685	2.3491	8
18	C4 H5 N6 O3 F3	242.0369	-0.5743	-2.3729	4

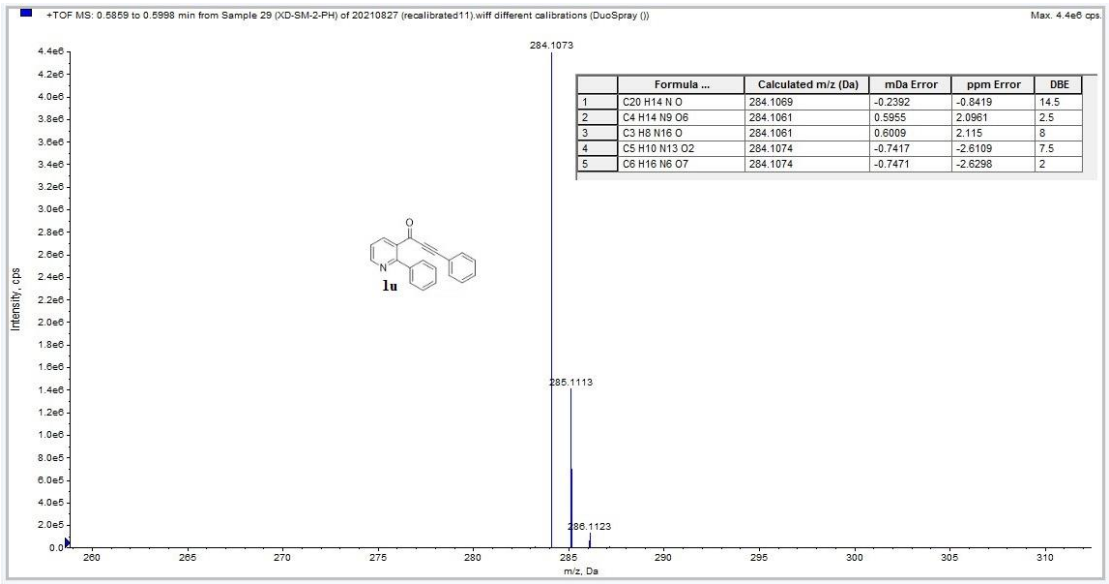



176.4519
149.7662
139.1885
133.3343
132.6536
131.3174
129.7500
129.6939
128.9175
127.8596
94.8254
86.6350
77.3195
77.1072
76.8951
1t
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

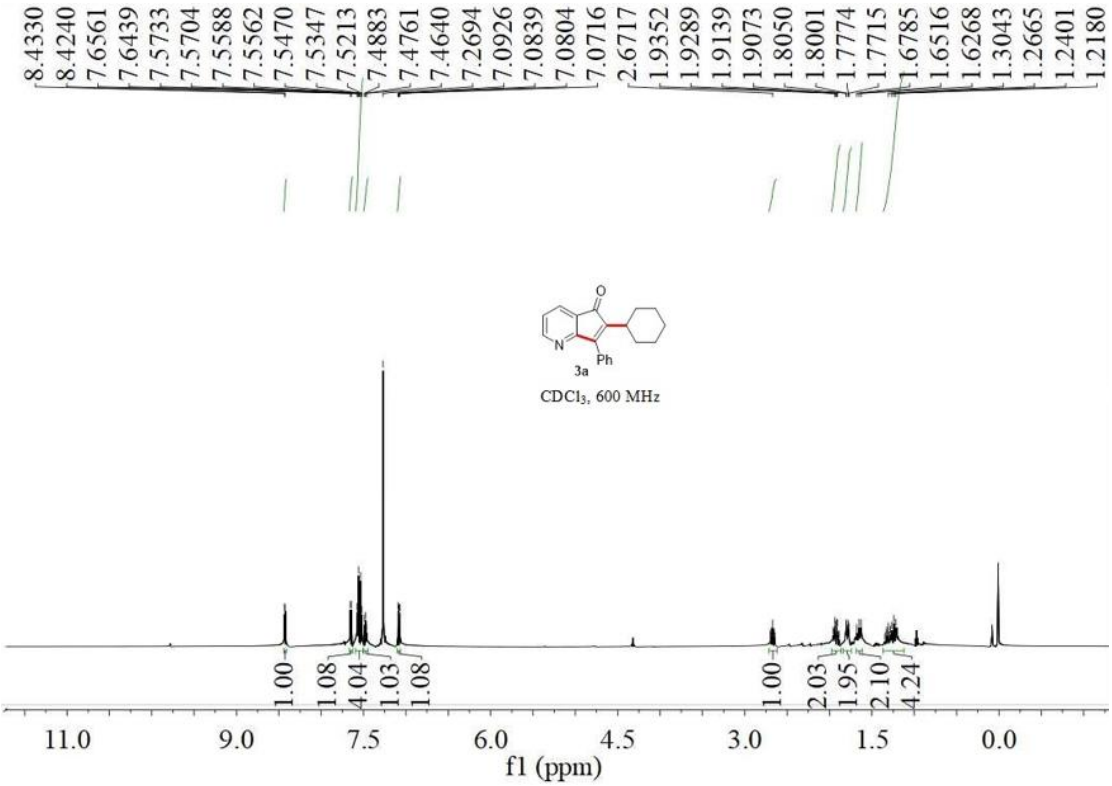

+TOF MS: 0.8137 to 0.8416 min from Sample 13 (XD-LIANBPY-SM) of 20210713 (recalibrated4).wiff different calibrations (DuoSpray ())
Max. 4.4e6 cps
285.1024
286.1058
287.1085
Intensity, cps
m/z, Da
1t
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C19 H13 N2 O | 285.1022 | 0.1604 | 0.5626 | 14.5
2 | C4 H9 N14 O2 | 285.1027 | -0.3421 | -1.2001 | 7.5
3 | C5 H15 N7 O7 | 285.1027 | -0.3475 | -1.219 | 2

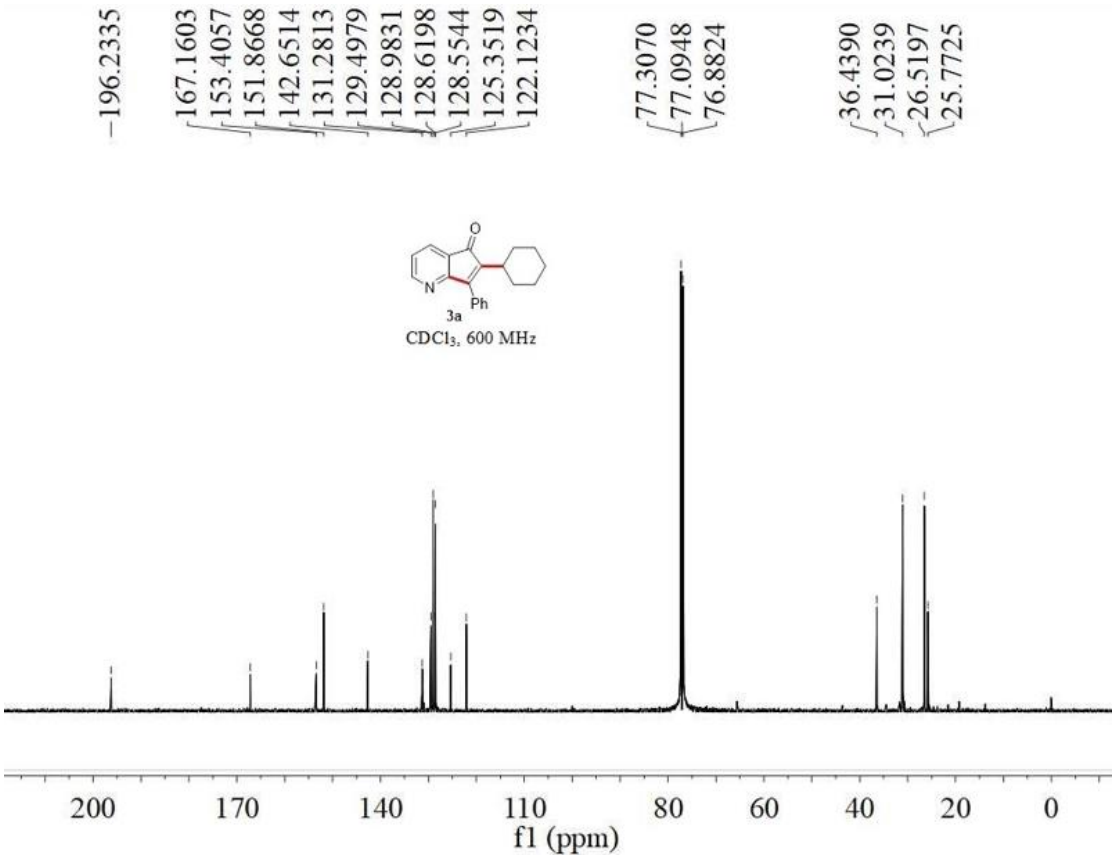

8.8028
8.7999
8.7949
8.7920
8.1871
8.1842
8.1741
8.1712
7.6711
7.6577
7.4611
7.4492
7.4364
7.4064
7.3941
7.3819
7.3704
7.3625
7.3574
7.3494
7.3448
7.3322
7.3197
7.2450
7.2317
7.2189
7.1705
7.1566
1u
CDCl3, 600 MHz
1.00
1.00
2.00
2.04
1.02
1.00
1.05
2.03
2.03
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

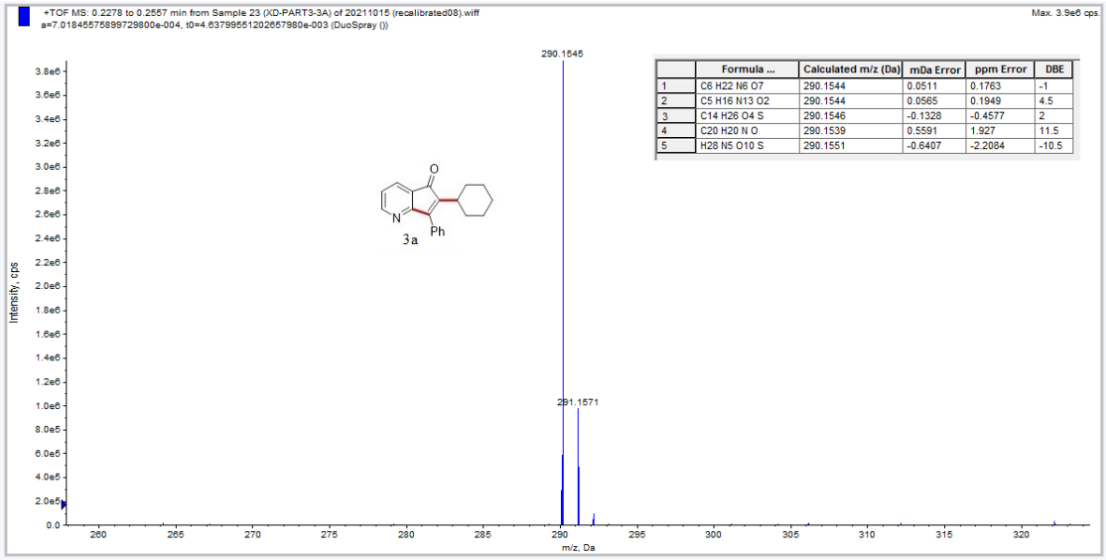

179.6762
159.3077
151.8845
137.8310
134.1392
133.1387
130.8637
130.0989
129.4644
128.5324
128.4487
121.9554
94.8779
88.5158
77.5212
77.3083
77.0958
1u
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

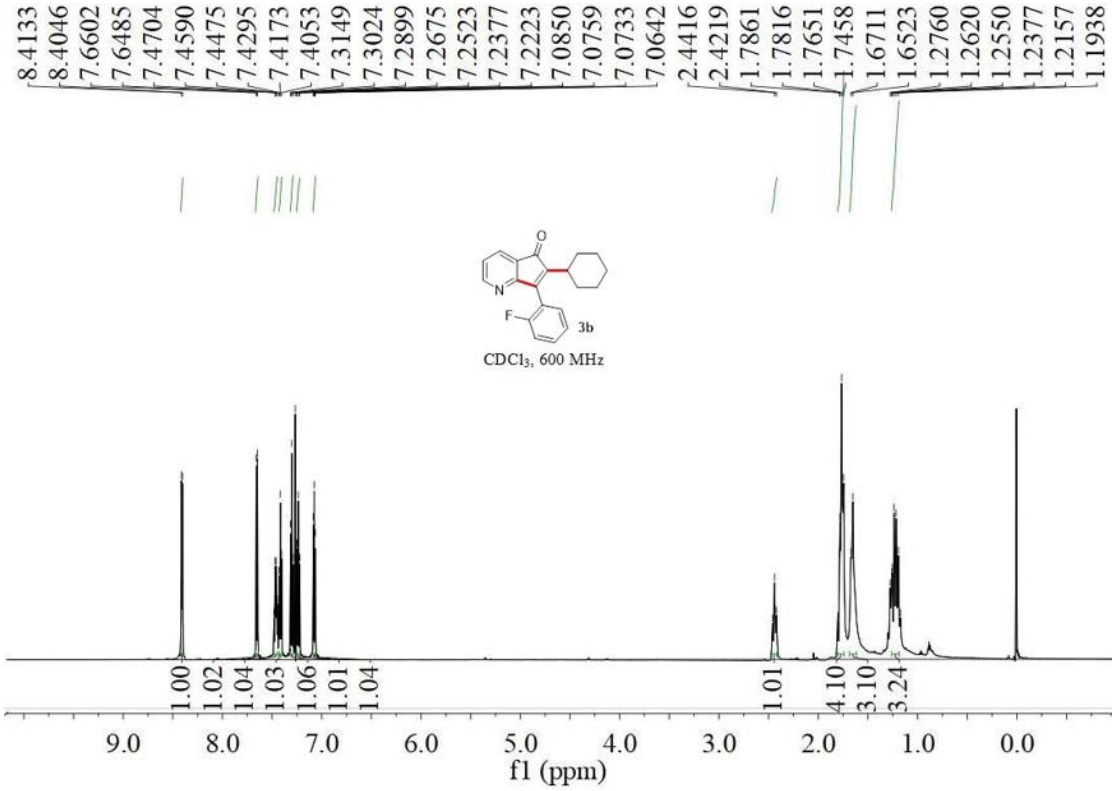


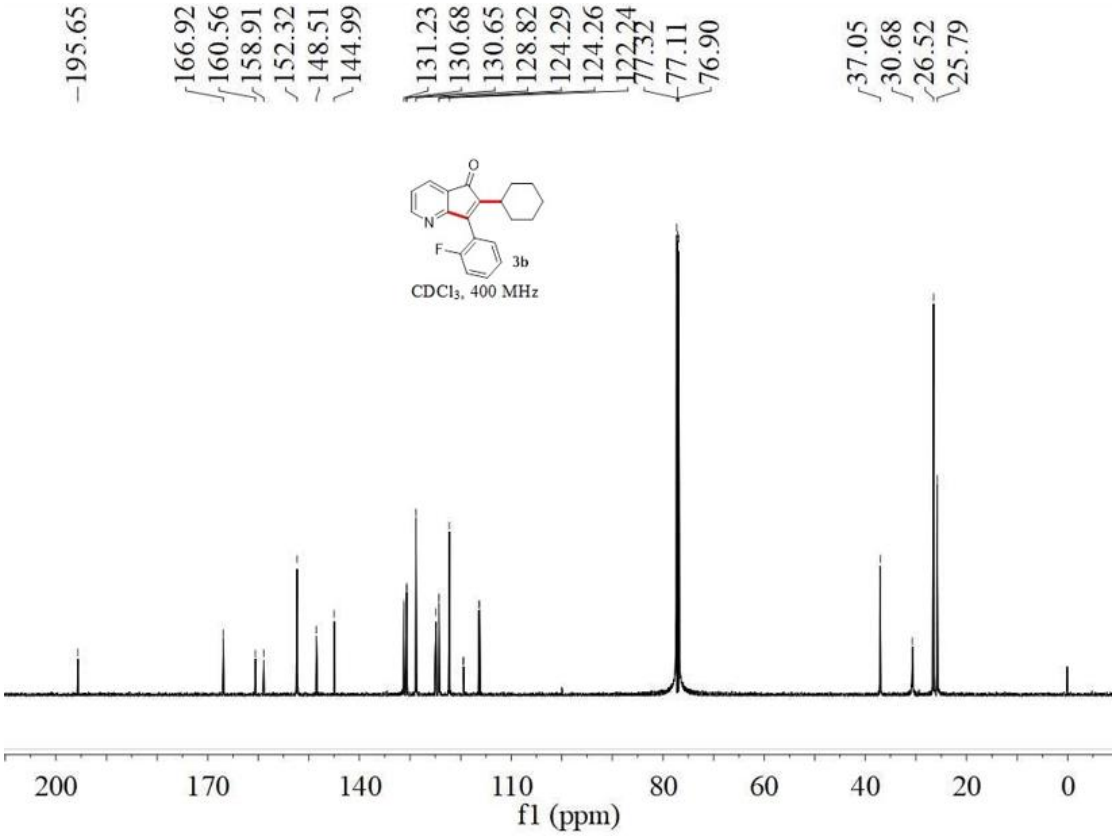
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C20 H14 N O	284.1069	-0.2392	-0.8419	14.5
2	C4 H14 N9 O6	284.1061	0.5955	2.0961	2.5
3	C3 H8 N16 O	284.1061	0.6009	2.115	8
4	C5 H10 N13 O2	284.1074	-0.7417	-2.6109	7.5
5	C6 H16 N6 O7	284.1074	-0.7471	-2.6298	2

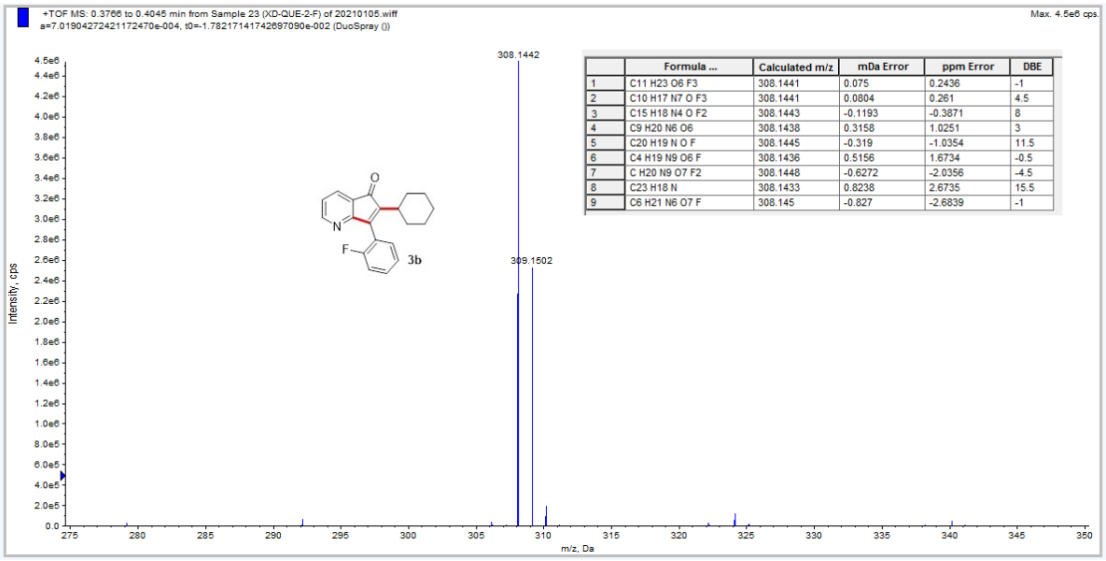



196.2335
167.1603
153.4057
151.8668
142.6514
131.2813
129.4979
128.9831
128.6198
128.5544
125.3519
122.1234
77.3070
77.0948
76.8824
36.4390
31.0239
26.5197
25.7725
O
N
Ph
3a
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

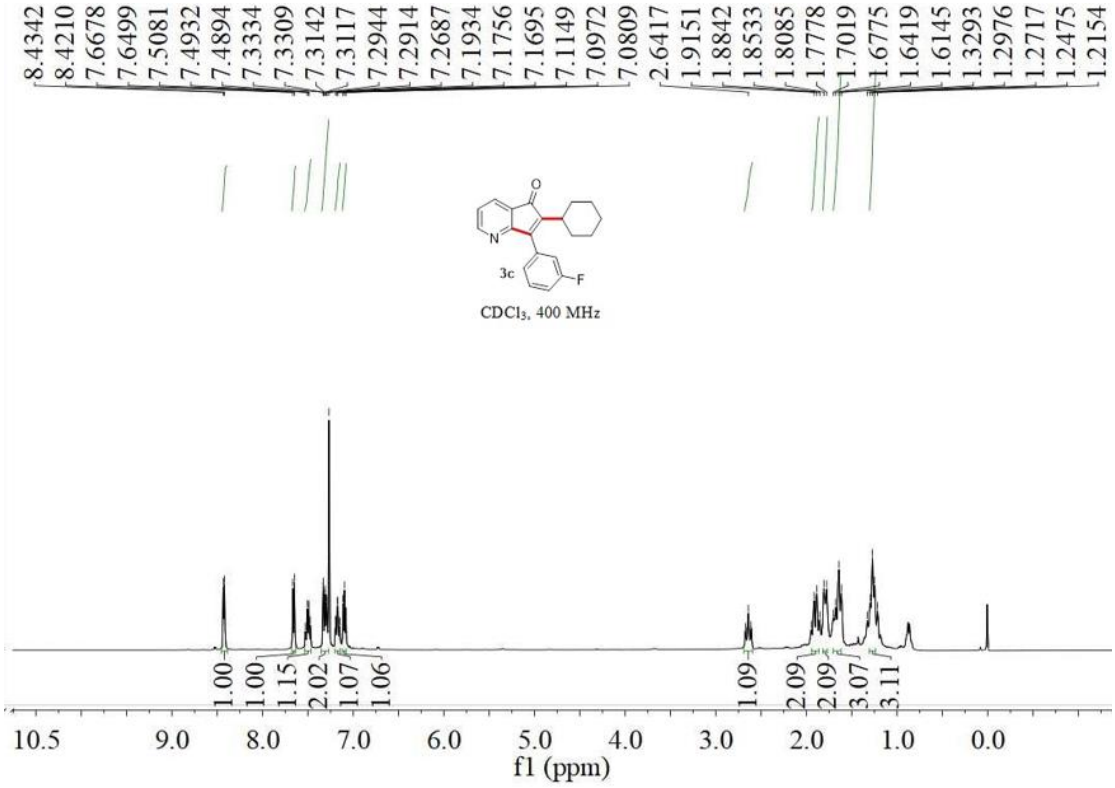

+TOF MS: 0.2278 to 0.2557 min from Sample 23 (XD-PART3-3A) of 20211015 (recalibrated08).wiff
a=7.018455758997298 00e-004, t0=4.637995512026579 80e-003 (DuoSpray ())
Max. 3.9e6 cps.
290.1545
291.1571
Intensity, cps
m/z, Da
O
N
Ph
3a
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C6 H22 N6 O7 | 290.1544 | 0.0511 | 0.1763 | -1
2 | C5 H16 N13 O2 | 290.1544 | 0.0565 | 0.1949 | 4.5
3 | C14 H26 O4 S | 290.1546 | -0.1328 | -0.4577 | 2
4 | C20 H20 N O | 290.1539 | 0.5591 | 1.927 | 11.5
5 | H28 N5 O10 S | 290.1551 | -0.6407 | -2.2084 | -10.5

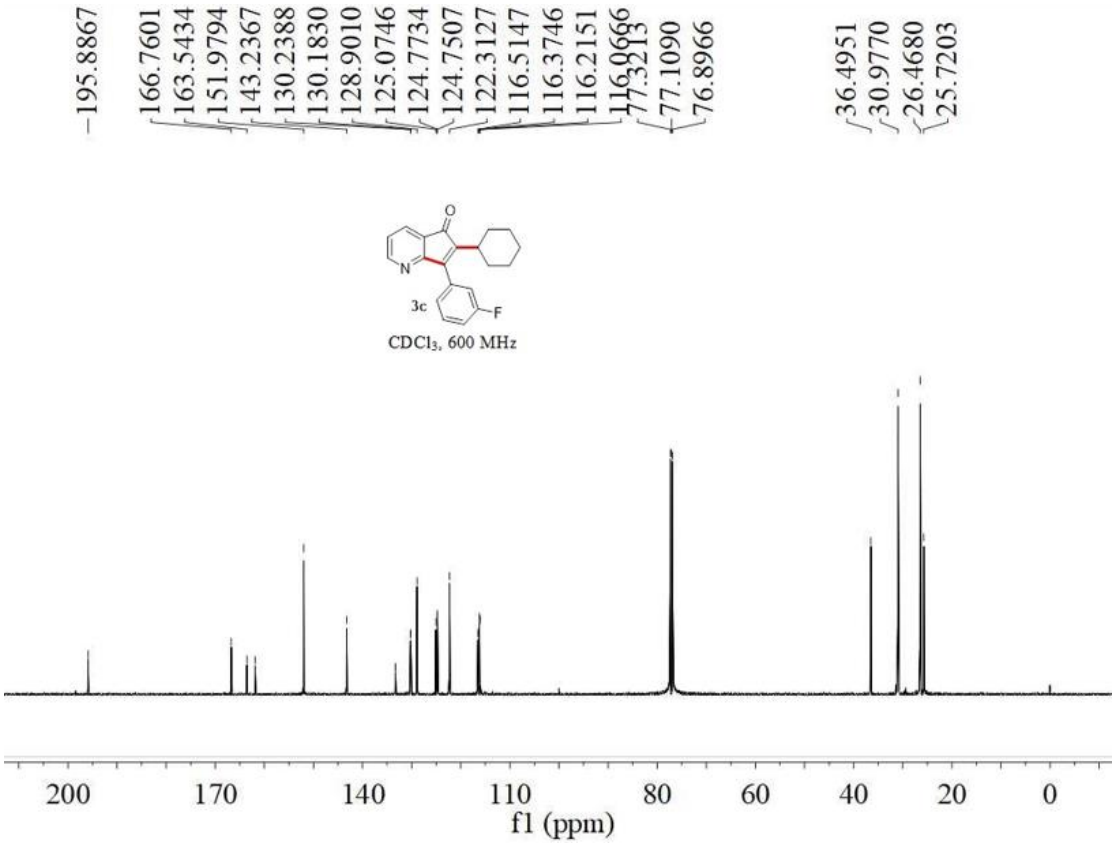

8.4133
8.4046
7.6602
7.6485
7.4704
7.4590
7.4475
7.4295
7.4173
7.4053
7.3149
7.3024
7.2899
7.2675
7.2523
7.2377
7.2223
7.0850
7.0759
7.0733
7.0642
2.4416
2.4219
1.7861
1.7816
1.7651
1.7458
1.6711
1.6523
1.2760
1.2620
1.2550
1.2377
1.2157
1.1938
O
N
F
3b
CDCl3, 600 MHz
1.00
1.02
1.04
1.03
1.06
1.01
1.04
1.01
4.10
3.10
3.24
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

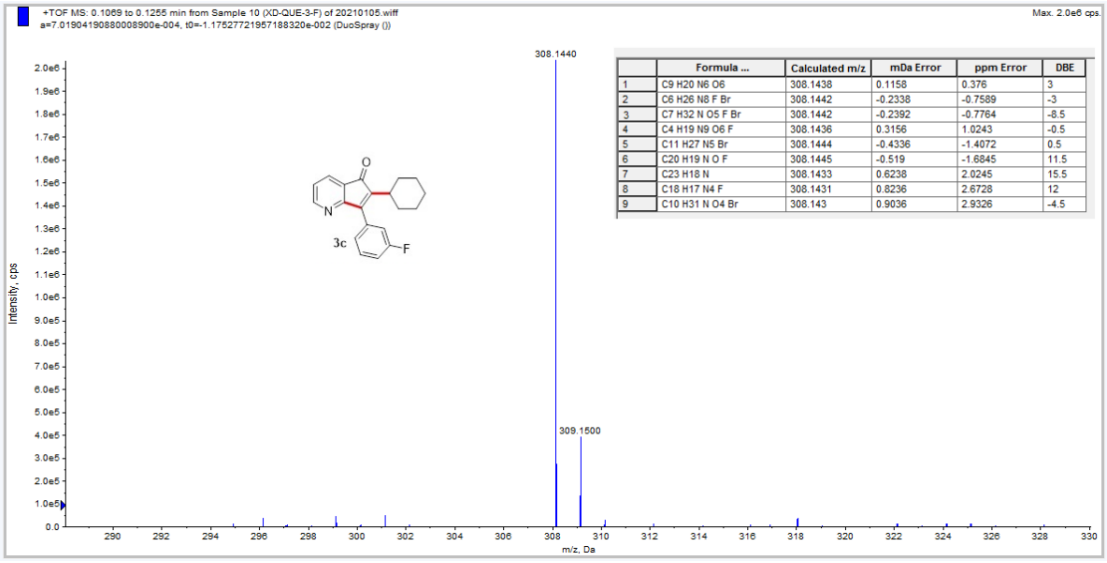

195.65
166.92
160.56
158.91
152.32
148.51
144.99
131.23
130.68
130.65
128.82
124.29
124.26
122.24
77.32
77.11
76.90
37.05
30.68
26.52
25.79
O
N
F
3b
CDCl3, 400 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

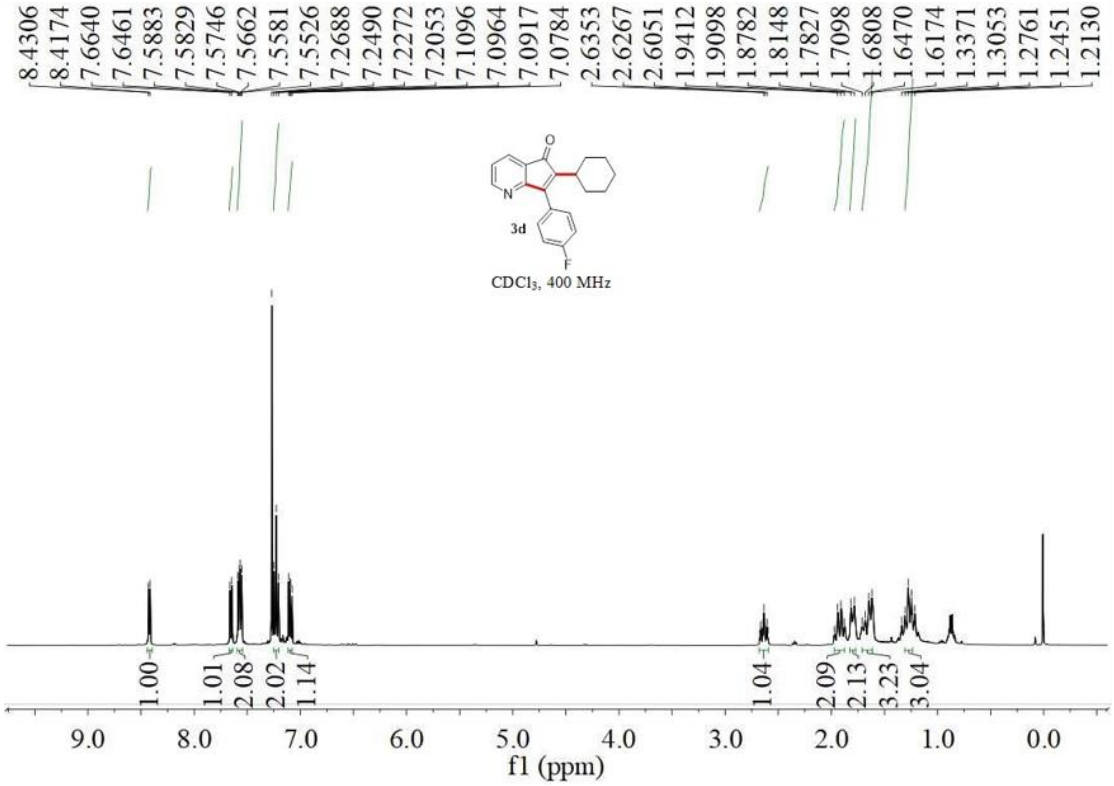


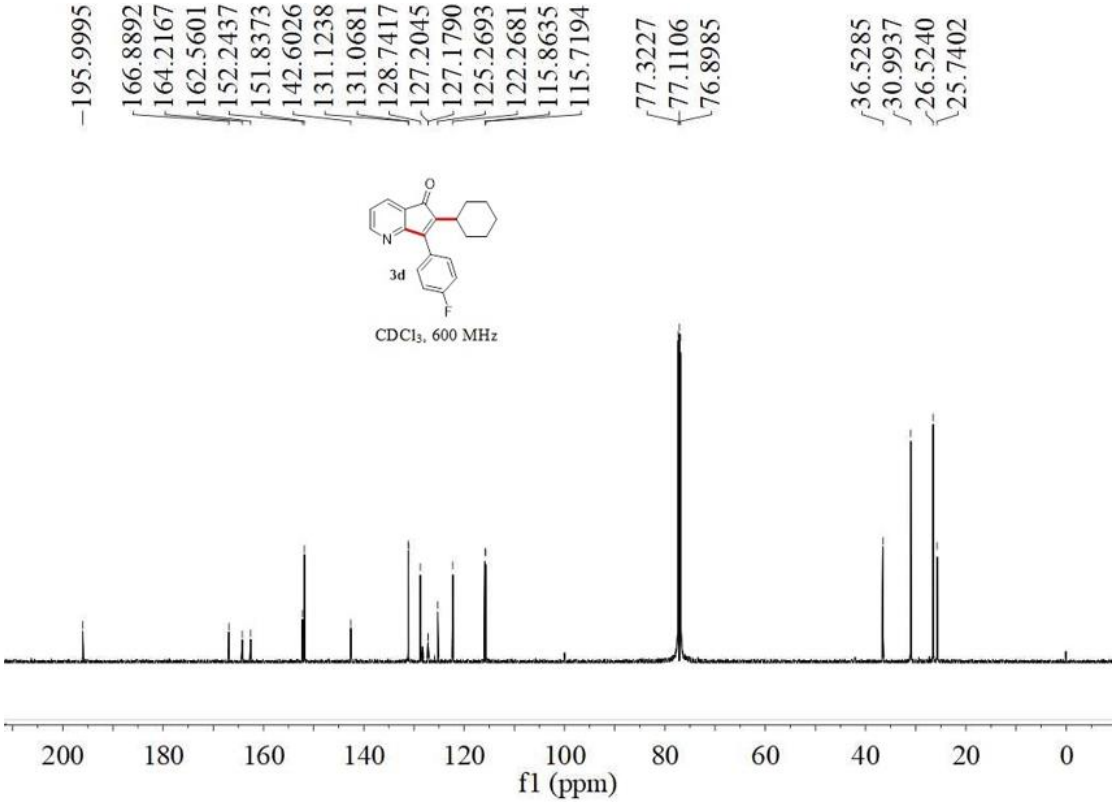
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C11 H23 O6 F3	308.1441	0.075	0.2436	-1
2	C10 H17 N7 O F3	308.1441	0.0804	0.261	4.5
3	C15 H18 N4 O F2	308.1443	-0.1193	-0.3871	8
4	C9 H20 N6 O6	308.1438	0.3158	1.0251	3
5	C20 H19 N O F	308.1445	-0.319	-1.0354	11.5
6	C4 H19 N9 O6 F	308.1436	0.5156	1.6734	-0.5
7	C H20 N9 O7 F2	308.1448	-0.6272	-2.0356	-4.5
8	C23 H18 N	308.1433	0.8238	2.6735	15.5
9	C6 H21 N6 O7 F	308.145	-0.827	-2.6839	-1

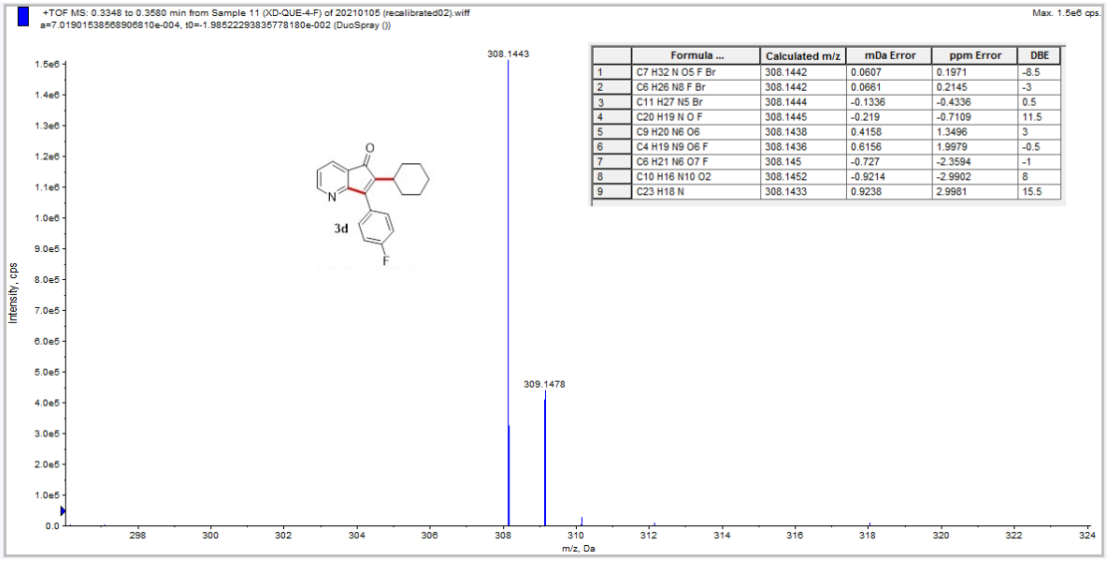



195.8867
166.7601
163.5434
151.9794
143.2367
130.2388
130.1830
128.9010
125.0746
124.7734
124.7507
122.3127
116.5147
116.3746
116.2151
116.0666
77.3213
77.1090
76.8966
36.4951
30.9770
26.4680
25.7203
O
N
3c
F
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

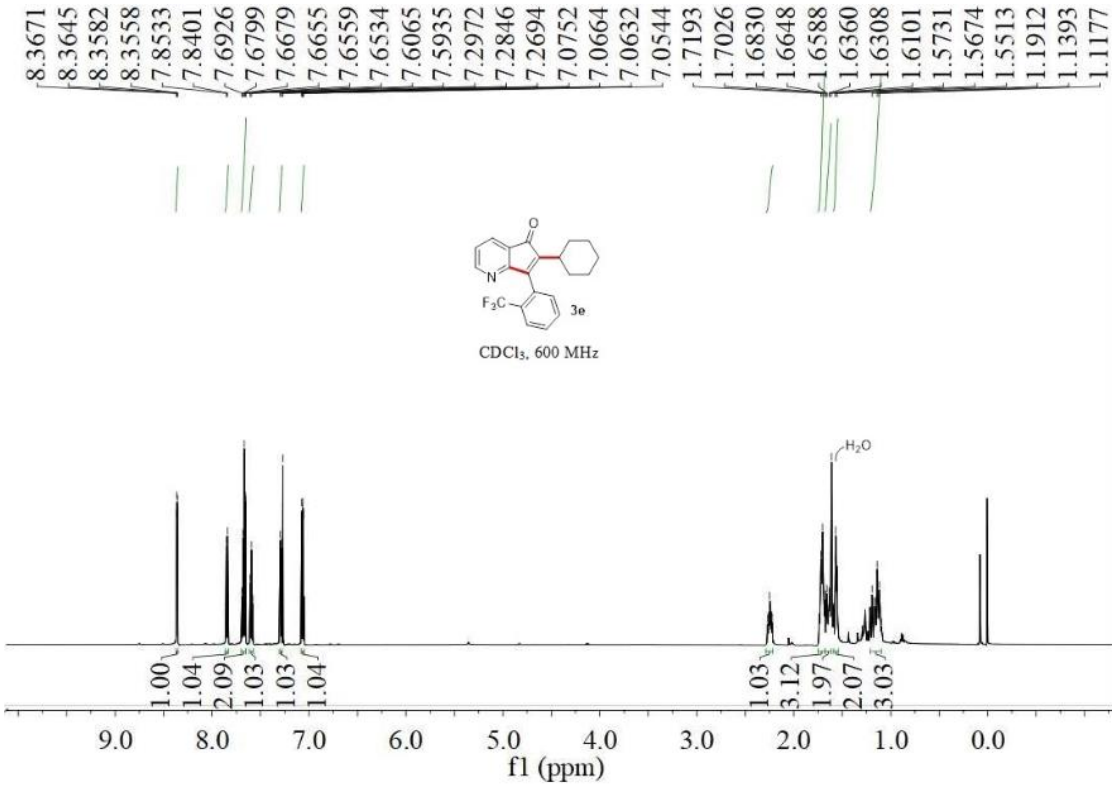

+TOF MS: 0.1069 to 0.1255 min from Sample 10 (XD-QUE-3-F) of 20210105.wiff
a=7.019041908800089e-004, t0=-1.1752772195718832e-002 (DuoSpray ())
Max. 2.0e6 cps.
308.1440
309.1500
Intensity, cps
m/z, Da
3c
F
N
O
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C9 H20 N6 O6 | 308.1438 | 0.1158 | 0.376 | 3
2 | C6 H26 N8 F Br | 308.1442 | -0.2338 | -0.7589 | -3
3 | C7 H32 N O5 F Br | 308.1442 | -0.2392 | -0.7764 | -8.5
4 | C4 H19 N9 O6 F | 308.1436 | 0.3156 | 1.0243 | -0.5
5 | C11 H27 N5 Br | 308.1444 | -0.4336 | -1.4072 | 0.5
6 | C20 H19 N O F | 308.1445 | -0.519 | -1.6845 | 11.5
7 | C23 H18 N | 308.1433 | 0.6238 | 2.0245 | 15.5
8 | C18 H17 N4 F | 308.1431 | 0.8236 | 2.6728 | 12
9 | C10 H31 N O4 Br | 308.143 | 0.9036 | 2.9326 | -4.5

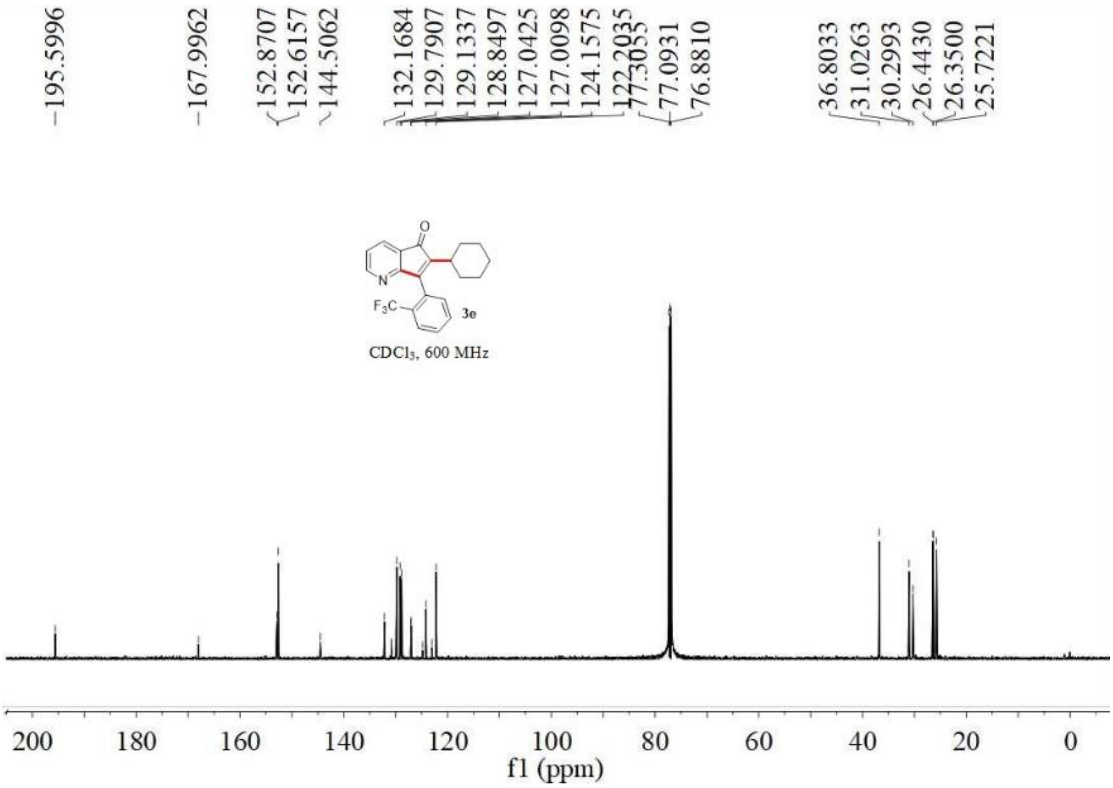

8.4306
8.4174
7.6640
7.6461
7.5883
7.5829
7.5746
7.5662
7.5581
7.5526
7.2688
7.2490
7.2272
7.2053
7.1096
7.0964
7.0917
7.0784
2.6353
2.6267
2.6051
1.9412
1.9098
1.8782
1.8148
1.7827
1.7098
1.6808
1.6470
1.6174
1.3371
1.3053
1.2761
1.2451
1.2130
3d
CDCl3, 400 MHz
1.00
1.01
2.08
2.02
1.14
1.04
2.09
2.13
3.23
3.04
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

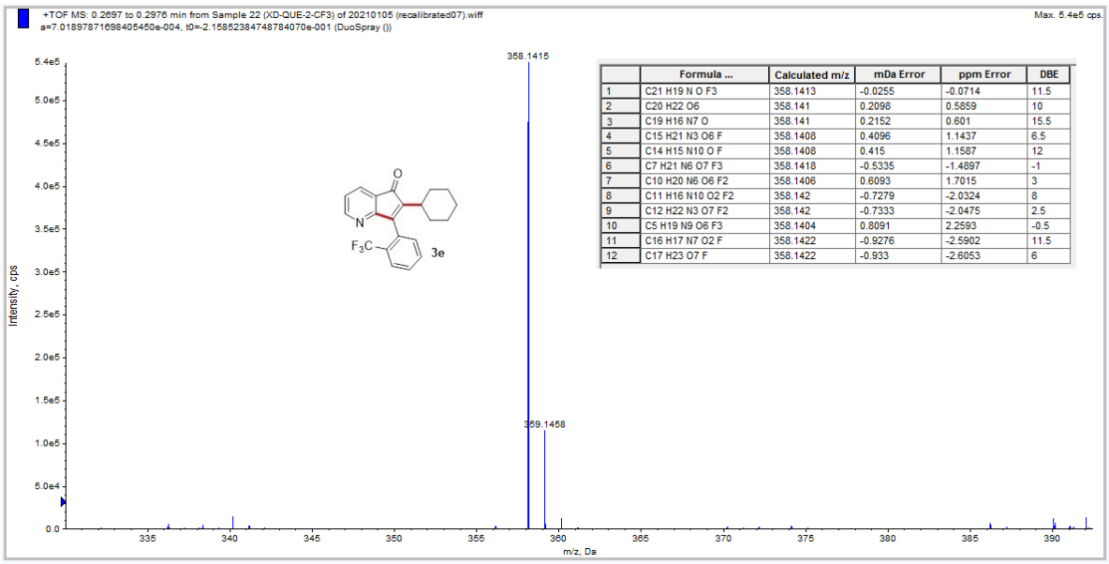

195.9995
166.8892
164.2167
162.5601
152.2437
151.8373
142.6026
131.1238
131.0681
128.7417
127.2045
127.1790
125.2693
122.2681
115.8635
115.7194
77.3227
77.1106
76.8985
36.5285
30.9937
26.5240
25.7402
3d
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



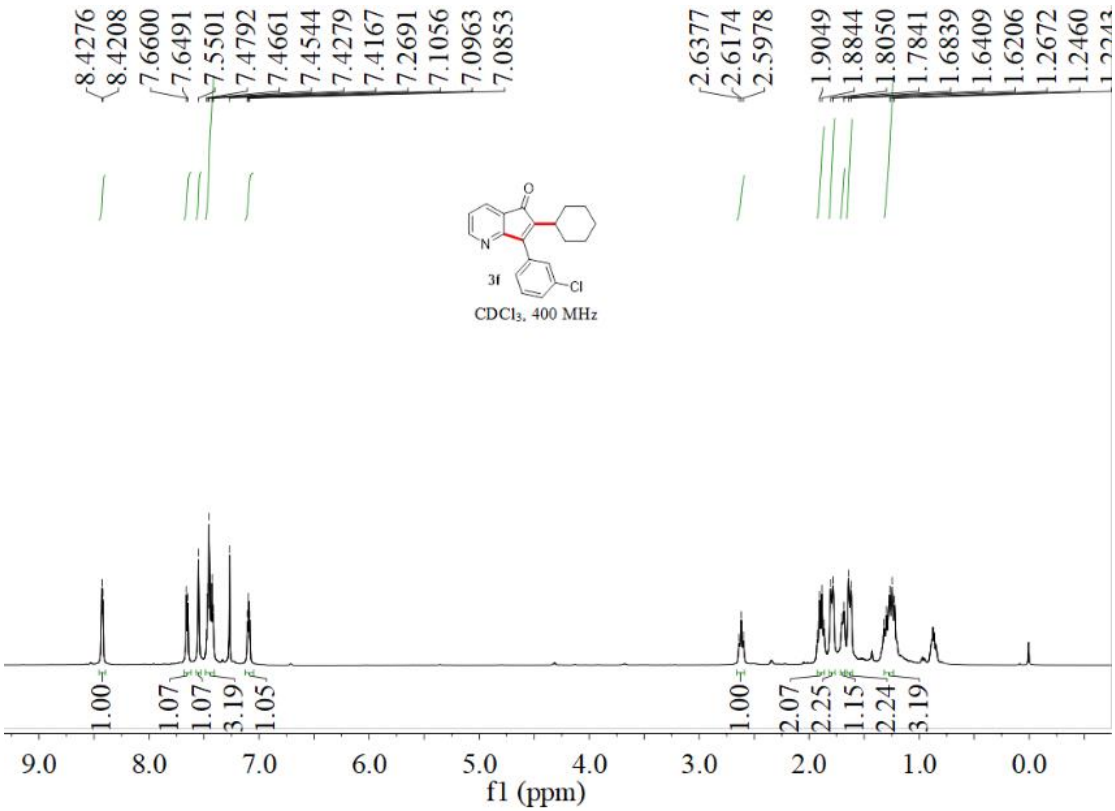
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C7 H32 N O5 F Br	308.1442	0.0607	0.1971	-8.5
2	C6 H26 N8 F Br	308.1442	0.0661	0.2145	-3
3	C11 H27 N5 Br	308.1444	-0.1336	-0.4336	0.5
4	C20 H19 N O F	308.1445	-0.219	-0.7109	11.5
5	C9 H20 N6 O6	308.1438	0.4158	1.3496	3
6	C4 H19 N9 O6 F	308.1436	0.6156	1.9979	-0.5
7	C6 H21 N6 O7 F	308.145	-0.727	-2.3594	-1
8	C10 H16 N10 O2	308.1452	-0.9214	-2.9902	8
9	C23 H18 N	308.1433	0.9238	2.9981	15.5

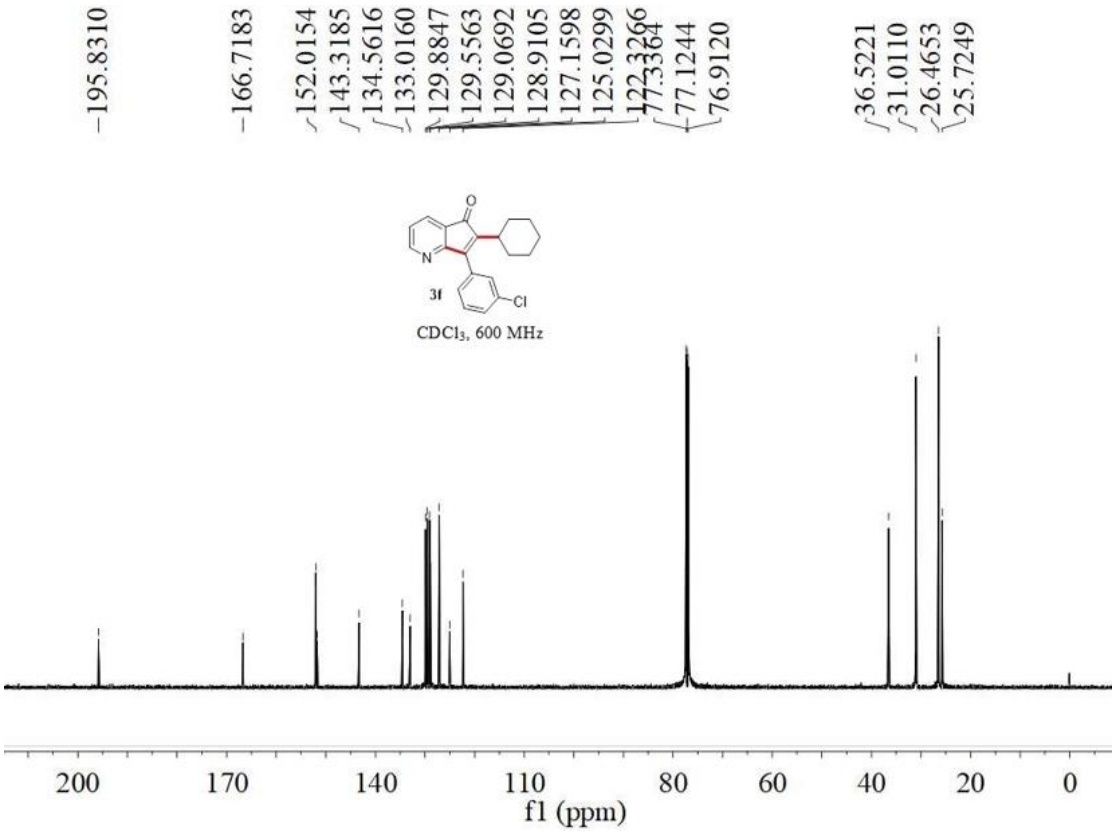


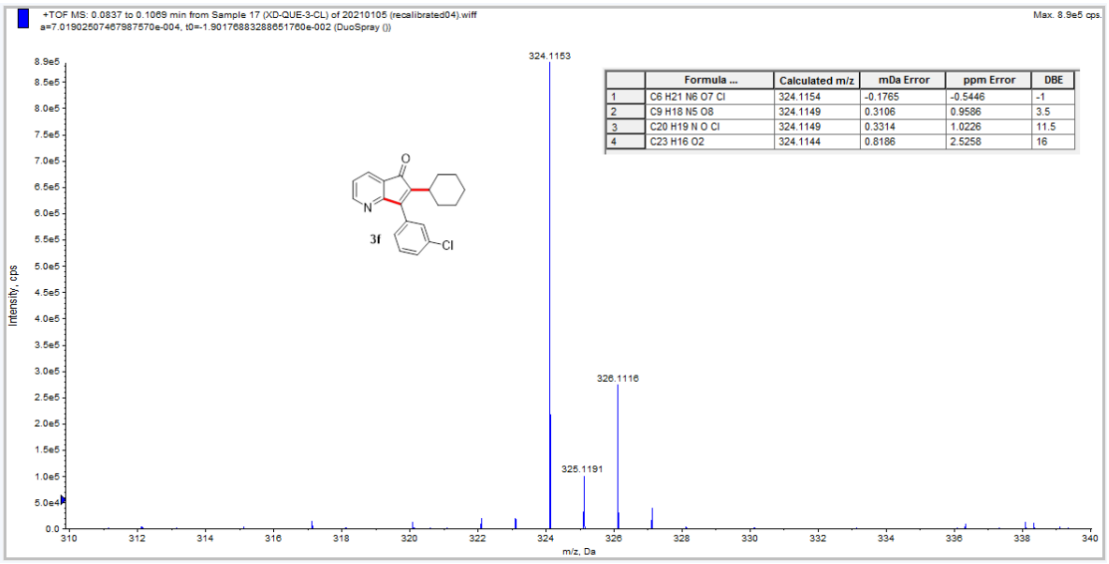


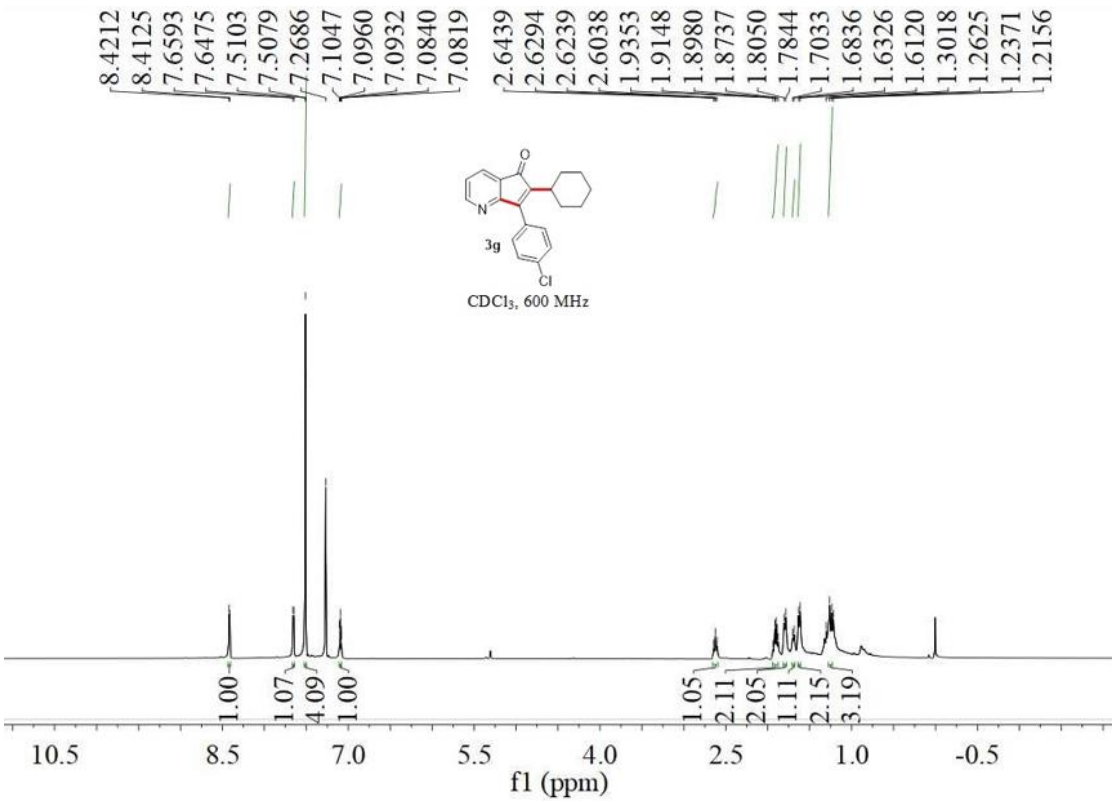


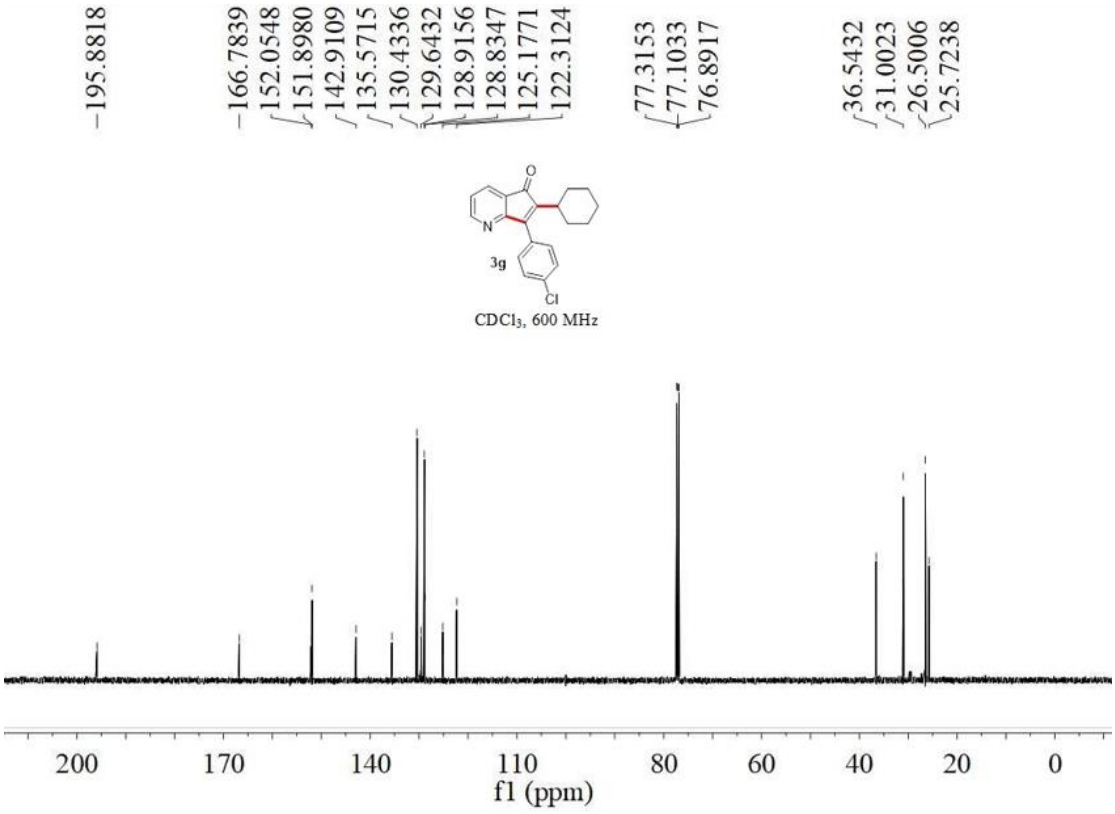
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C21 H19 N O F3	358.1413	-0.0255	-0.0714	11.5
2	C20 H22 O6	358.141	0.2098	0.5859	10
3	C19 H16 N7 O	358.141	0.2152	0.601	15.5
4	C15 H21 N3 O6 F	358.1408	0.4096	1.1437	6.5
5	C14 H15 N10 O F	358.1408	0.415	1.1587	12
6	C7 H21 N6 O7 F3	358.1418	-0.5335	-1.4897	-1
7	C10 H20 N6 O6 F2	358.1406	0.6093	1.7015	3
8	C11 H16 N10 O2 F2	358.142	-0.7279	-2.0324	8
9	C12 H22 N3 O7 F2	358.142	-0.7333	-2.0475	2.5
10	C5 H19 N9 O6 F3	358.1404	0.8091	2.2593	-0.5
11	C16 H17 N7 O2 F	358.1422	-0.9276	-2.5902	11.5
12	C17 H23 O7 F	358.1422	-0.933	-2.6053	6

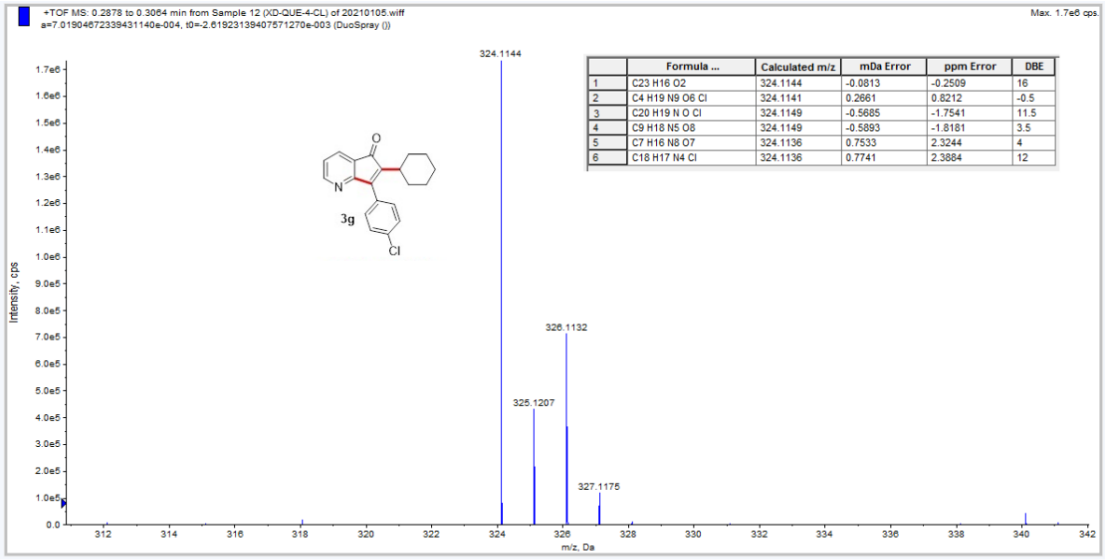

8.4276
8.4208
7.6600
7.6491
7.5501
7.4792
7.4661
7.4544
7.4279
7.4167
7.2691
7.1056
7.0963
7.0853
2.6377
2.6174
2.5978
1.9049
1.8844
1.8050
1.7841
1.6839
1.6409
1.6206
1.2672
1.2460
O
N
3f
Cl
CDCl3, 400 MHz
1.00
1.07
1.07
3.19
1.05
1.00
2.07
2.25
1.15
2.24
3.19
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

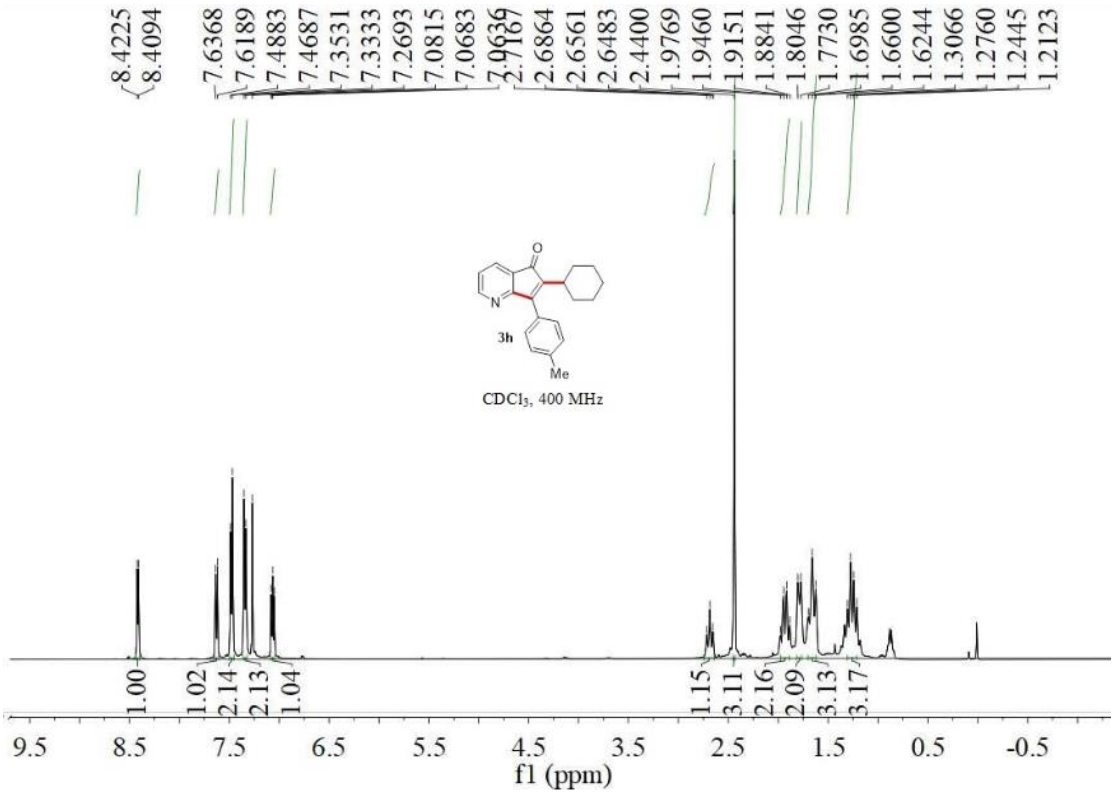

195.8310
166.7183
152.0154
143.3185
134.5616
133.0160
129.8847
129.5563
129.0692
128.9105
127.1598
125.0299
122.3266
77.3364
77.1244
76.9120
36.5221
31.0110
26.4653
25.7249
O
N
3f
Cl
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

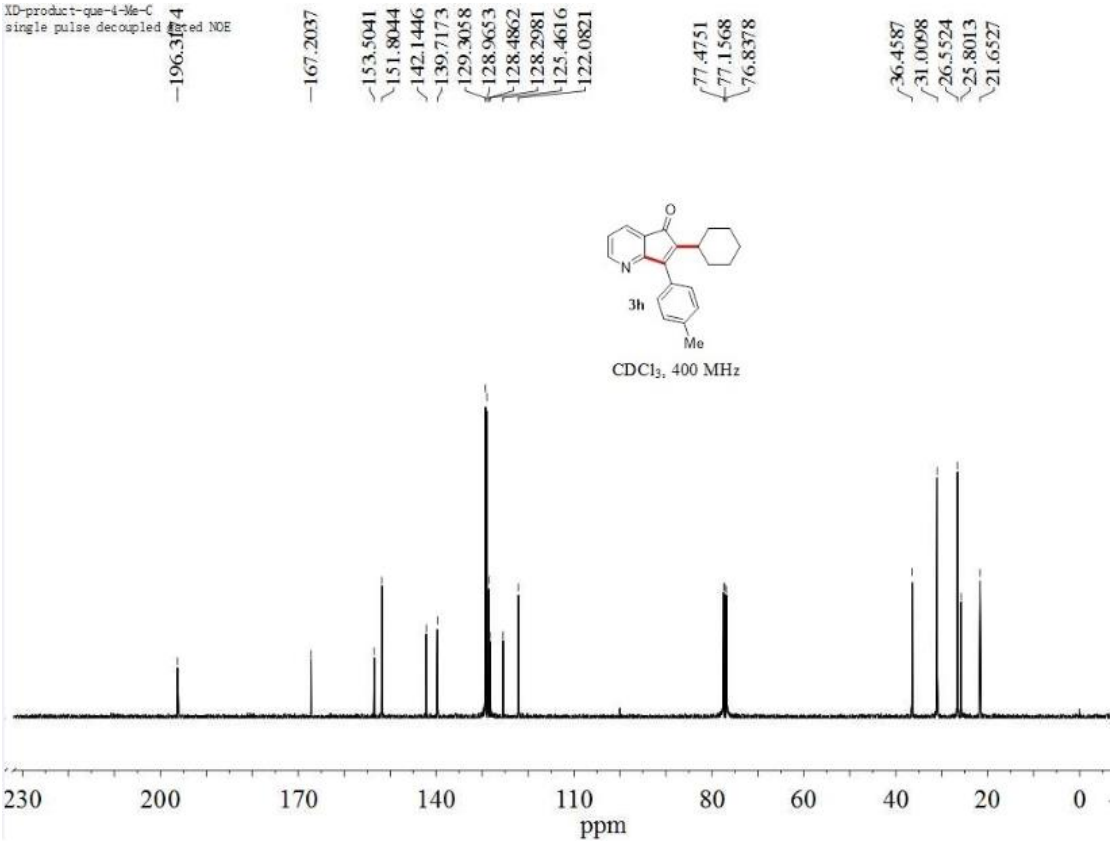

+TOF MS: 0.0837 to 0.1069 min from Sample 17 (XD-QUE-3-CL) of 20210105 (recalibrated04).wiff
a=7.019025074679875 70e-004, t0=-1.901768832886517 60e-002 (DuoSpray ())
Max. 8.9e5 cps.
324.1153
326.1116
325.1191
3f
Cl
Intensity, cps
m/z, Da
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C6 H21 N6 O7 Cl | 324.1154 | -0.1765 | -0.5446 | -1
2 | C9 H18 N5 O8 | 324.1149 | 0.3106 | 0.9586 | 3.5
3 | C20 H19 N O Cl | 324.1149 | 0.3314 | 1.0226 | 11.5
4 | C23 H16 O2 | 324.1144 | 0.8186 | 2.5258 | 16

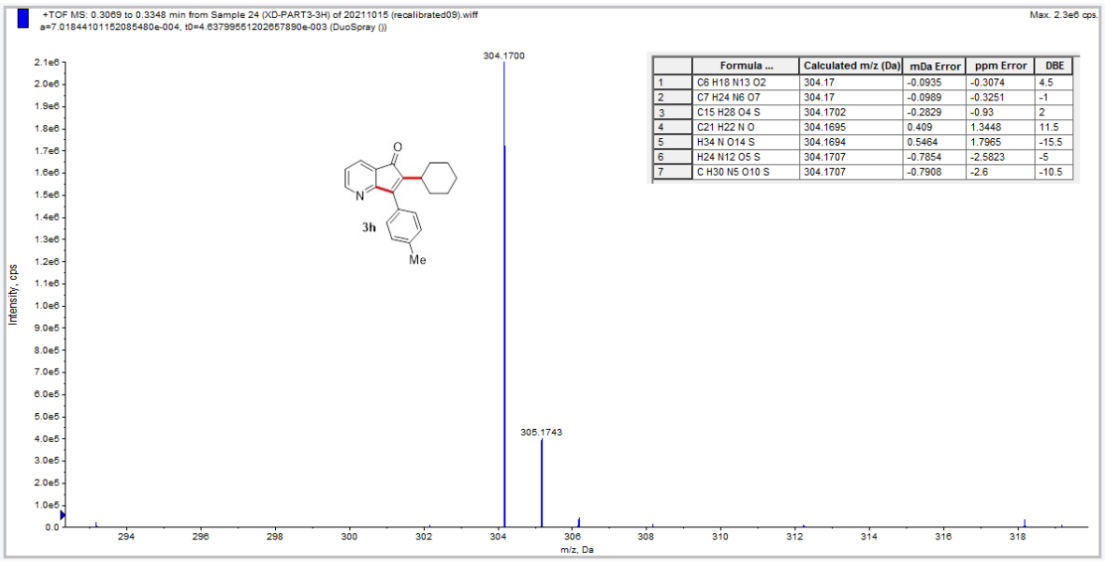

8.4212
8.4125
7.6593
7.6475
7.5103
7.5079
7.2686
7.1047
7.0960
7.0932
7.0840
7.0819
2.6439
2.6294
2.6239
2.6038
1.9353
1.9148
1.8980
1.8737
1.8050
1.7844
1.7033
1.6836
1.6326
1.6120
1.3018
1.2625
1.2371
1.2156
3g
Cl
CDCl3, 600 MHz
1.00
1.07
4.09
1.00
1.05
2.11
2.05
1.11
2.15
3.19
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

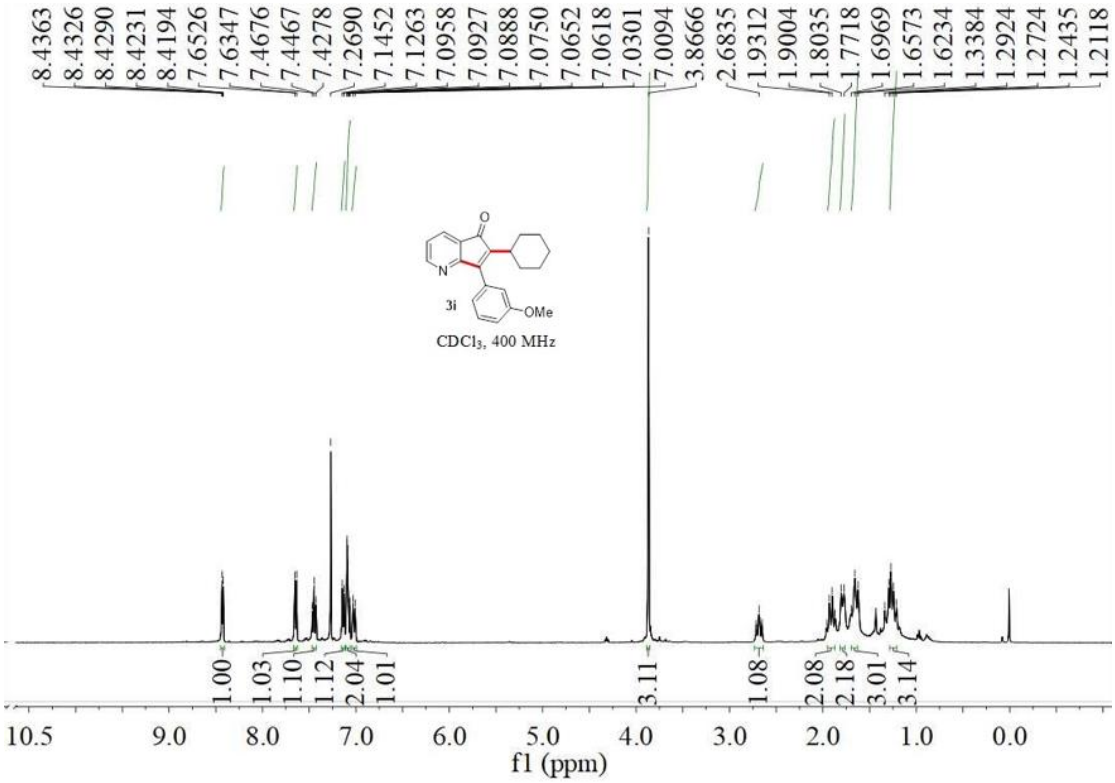

195.8818
166.7839
152.0548
151.8980
142.9109
135.5715
130.4336
129.6432
128.9156
128.8347
125.1771
122.3124
77.3153
77.1033
76.8917
36.5432
31.0023
26.5006
25.7238
O
N
3g
Cl
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

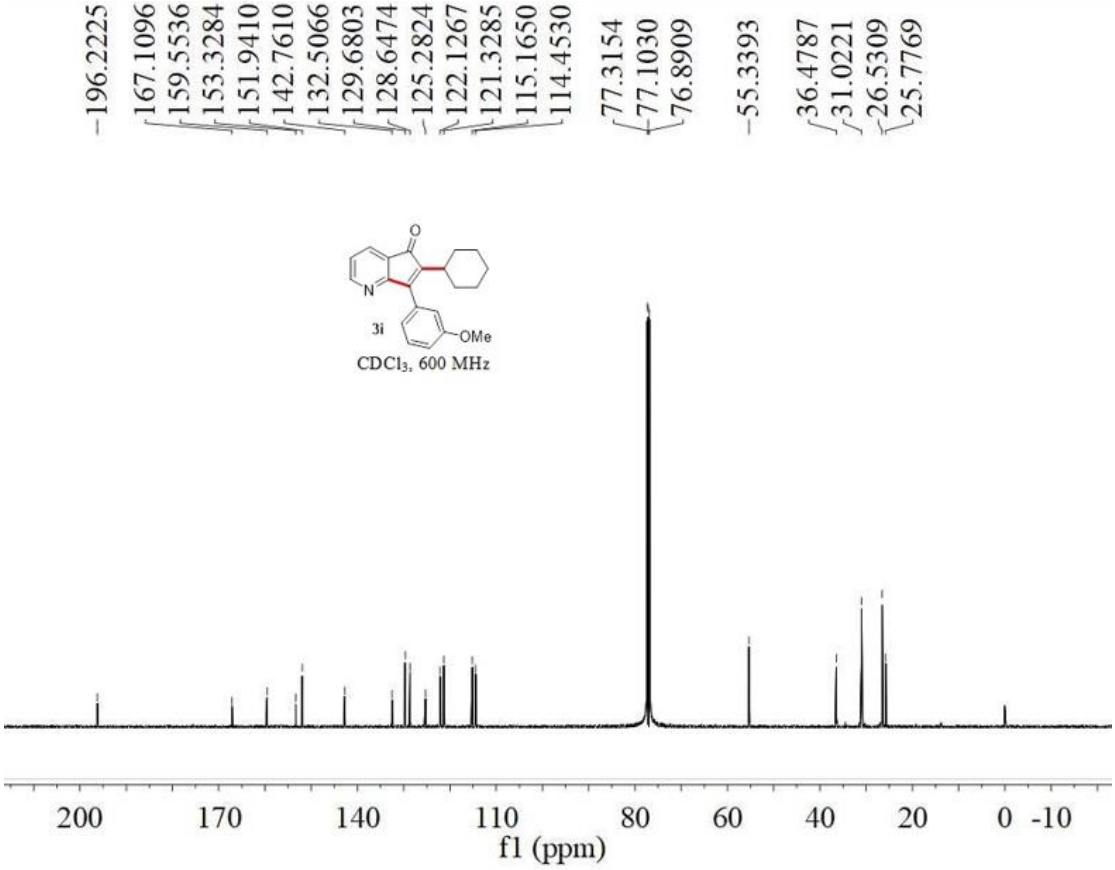

+TOF MS: 0.2878 to 0.3064 min from Sample 12 (XD-QUE-4-CL) of 20210105.wiff
a=7.0190467233943114 0e-004, t0=-2.6192313940757127 0e-003 (DuoSpray ())
Max. 1.7e6 cps
324.1144
326.1132
325.1207
327.1175
Intensity, cps
m/z, Da
3g
Cl
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C23 H16 O2 | 324.1144 | -0.0813 | -0.2509 | 16
2 | C4 H19 N9 O6 Cl | 324.1141 | 0.2661 | 0.8212 | -0.5
3 | C20 H19 N O Cl | 324.1149 | -0.5685 | -1.7541 | 11.5
4 | C9 H18 N5 O8 | 324.1149 | -0.5893 | -1.8181 | 3.5
5 | C7 H16 N8 O7 | 324.1136 | 0.7533 | 2.3244 | 4
6 | C18 H17 N4 Cl | 324.1136 | 0.7741 | 2.3884 | 12

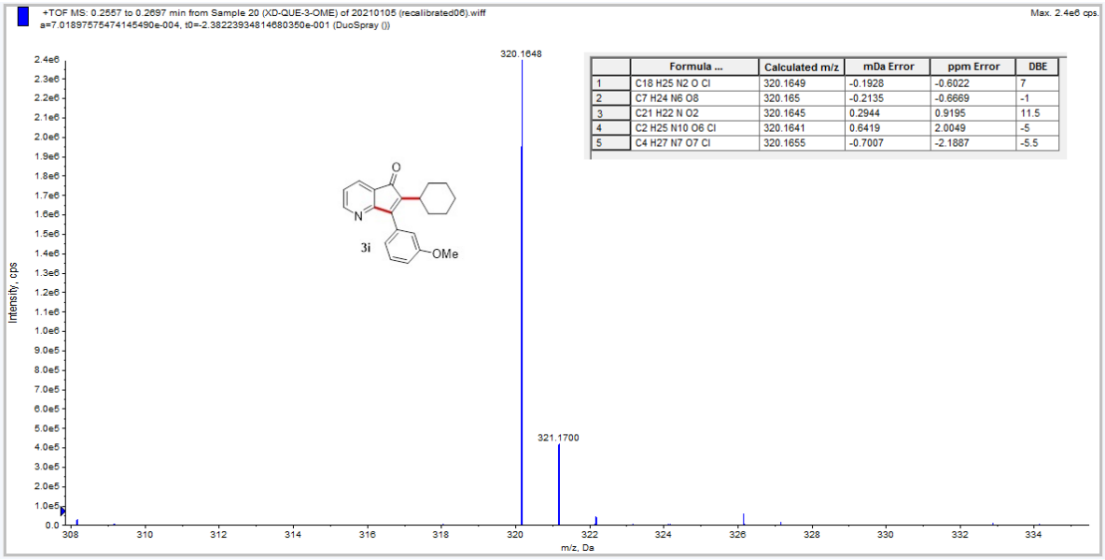

8.4225
8.4094
7.6368
7.6189
7.4883
7.4687
7.3531
7.3333
7.2693
7.0815
7.0683
7.9636
2.7167
2.6864
2.6561
2.6483
2.4400
1.9769
1.9460
1.9151
1.8841
1.8046
1.7730
1.6985
1.6600
1.6244
1.3066
1.2760
1.2445
1.2123
3h
Me
CDCl3, 400 MHz
1.00
1.02
2.14
2.13
1.04
1.15
3.11
2.16
2.09
3.13
3.17
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

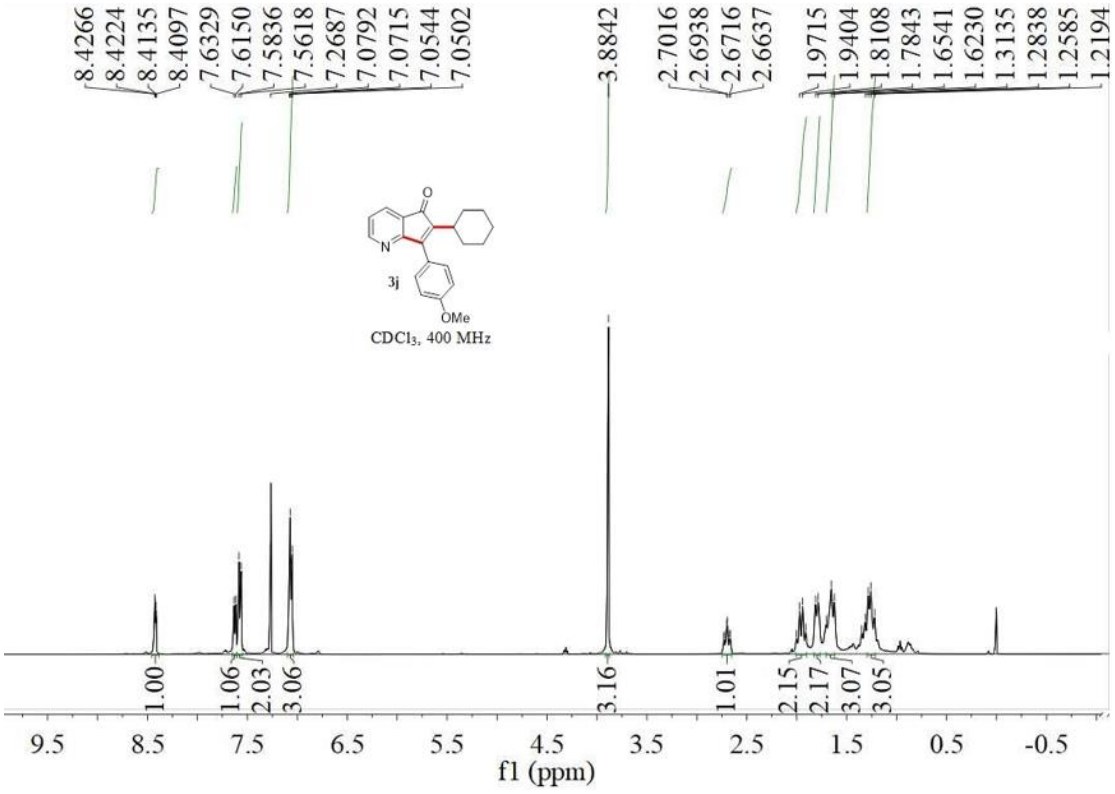

XD-product-que-4-Me-C
single pulse decoupled gated NOE
196.3764
167.2037
153.5041
151.8044
142.1446
139.7173
129.3058
128.9653
128.4862
128.2981
125.4616
122.0821
77.4751
77.1568
76.8378
36.4587
31.0098
26.5524
25.8013
21.6527
3h
Me
CDCl3, 400 MHz
230
200
170
140
110
80
60
40
20
0
ppm

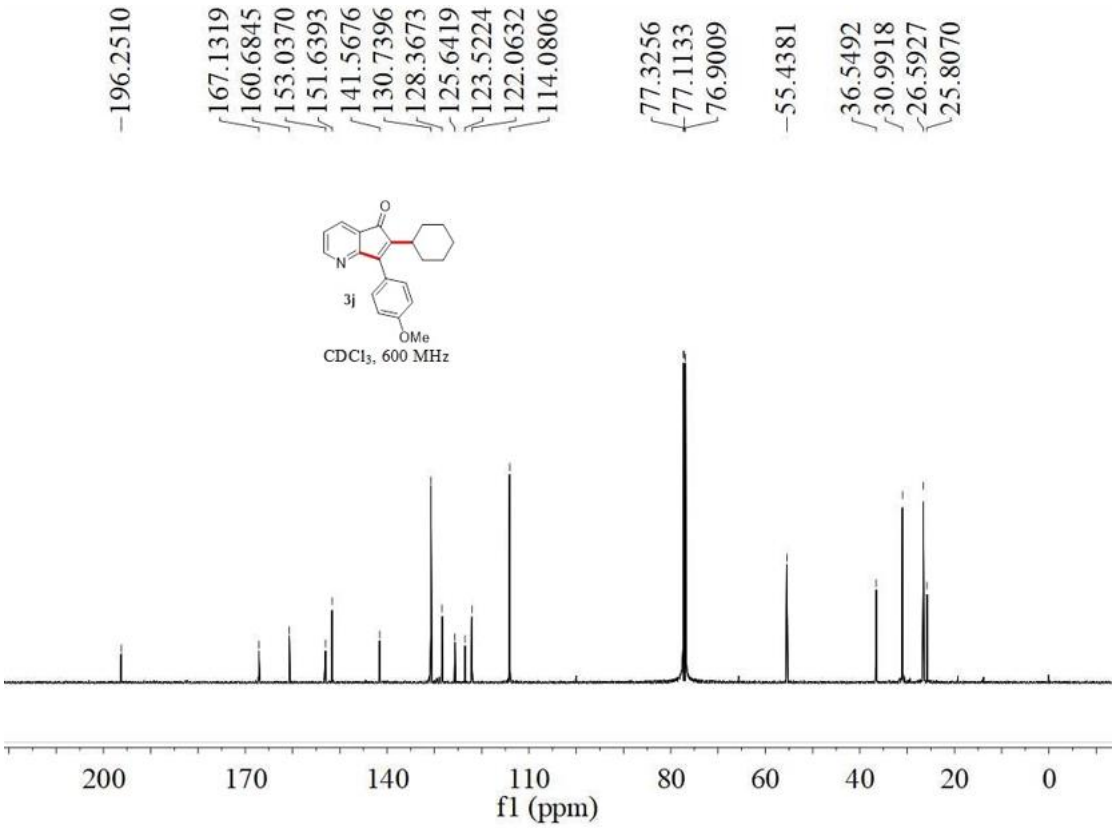

+TOF MS: 0.3069 to 0.3348 min from Sample 24 (XD-PART3-3H) of 20211015 (recalibrated09).wiff
a=7.0184410115208548 0e-004, t0=4.637995512026578 90e-003 (DuoSpray ())
Max. 2.3e6 cps.
304.1700
305.1743
Intensity, cps
m/z, Da
3h
Me
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C6 H18 N13 O2 | 304.17 | -0.0935 | -0.3074 | 4.5
2 | C7 H24 N6 O7 | 304.17 | -0.0989 | -0.3251 | -1
3 | C15 H28 O4 S | 304.1702 | -0.2829 | -0.93 | 2
4 | C21 H22 N O | 304.1695 | 0.409 | 1.3448 | 11.5
5 | H34 N O14 S | 304.1694 | 0.5464 | 1.7965 | -15.5
6 | H24 N12 O5 S | 304.1707 | -0.7854 | -2.5823 | -5
7 | C H30 N5 O10 S | 304.1707 | -0.7908 | -2.6 | -10.5

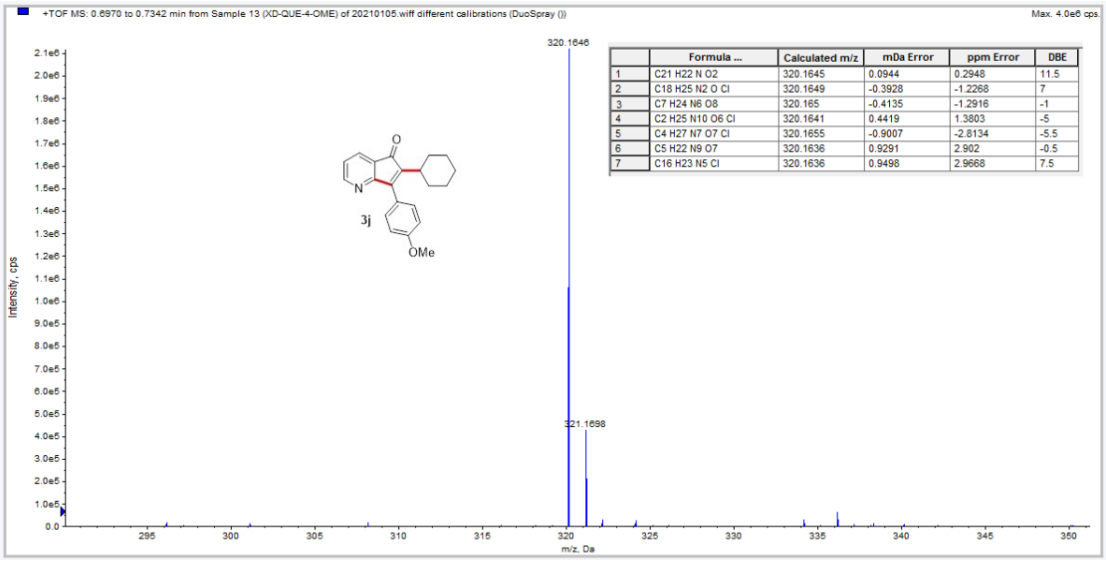

8.4363
8.4326
8.4290
8.4231
8.4194
7.6526
7.6347
7.4676
7.4467
7.4278
7.2690
7.1452
7.1263
7.0958
7.0927
7.0888
7.0750
7.0652
7.0618
7.0301
7.0094
3.8666
2.6835
1.9312
1.9004
1.8035
1.7718
1.6969
1.6573
1.6234
1.3384
1.2924
1.2724
1.2435
1.2118
3i
OMe
CDCl3, 400 MHz
1.00
1.03
1.10
1.12
2.04
1.01
3.11
1.08
2.08
2.18
3.01
3.14
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

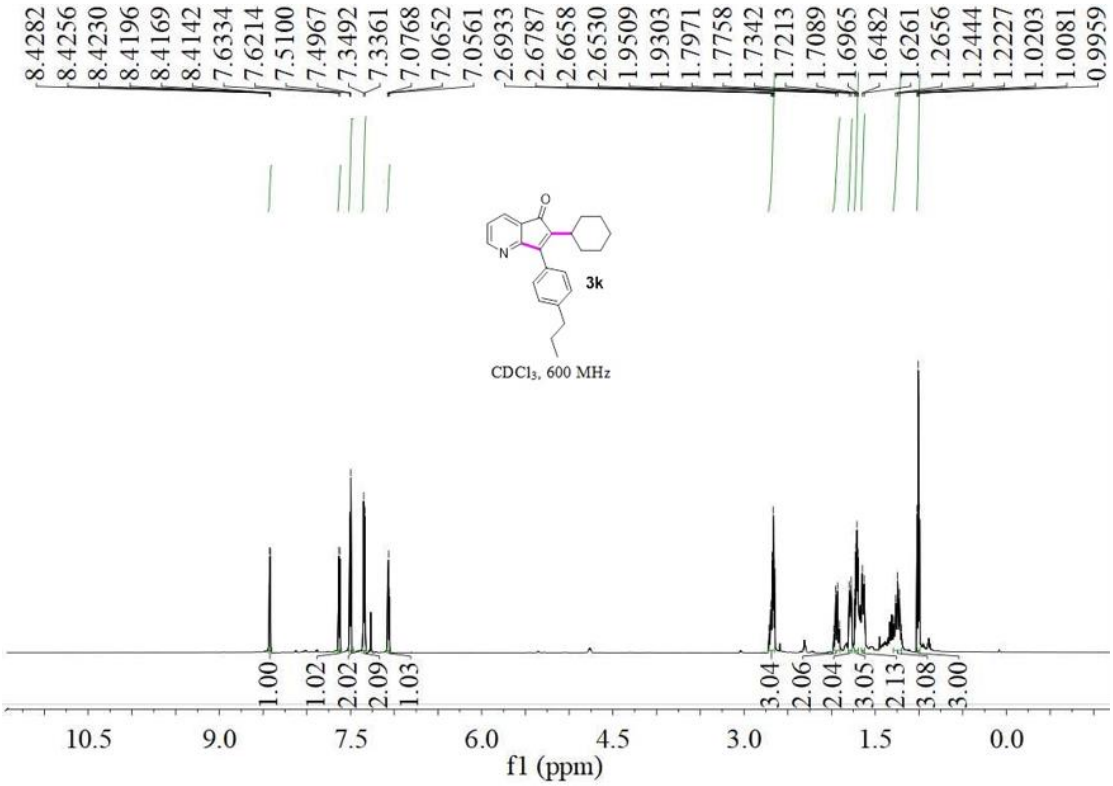

196.2225
167.1096
159.5536
153.3284
151.9410
142.7610
132.5066
129.6803
128.6474
125.2824
122.1267
121.3285
115.1650
114.4530
77.3154
77.1030
76.8909
55.3393
36.4787
31.0221
26.5309
25.7769
O
N
3i
OMe
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
-10
f1 (ppm)

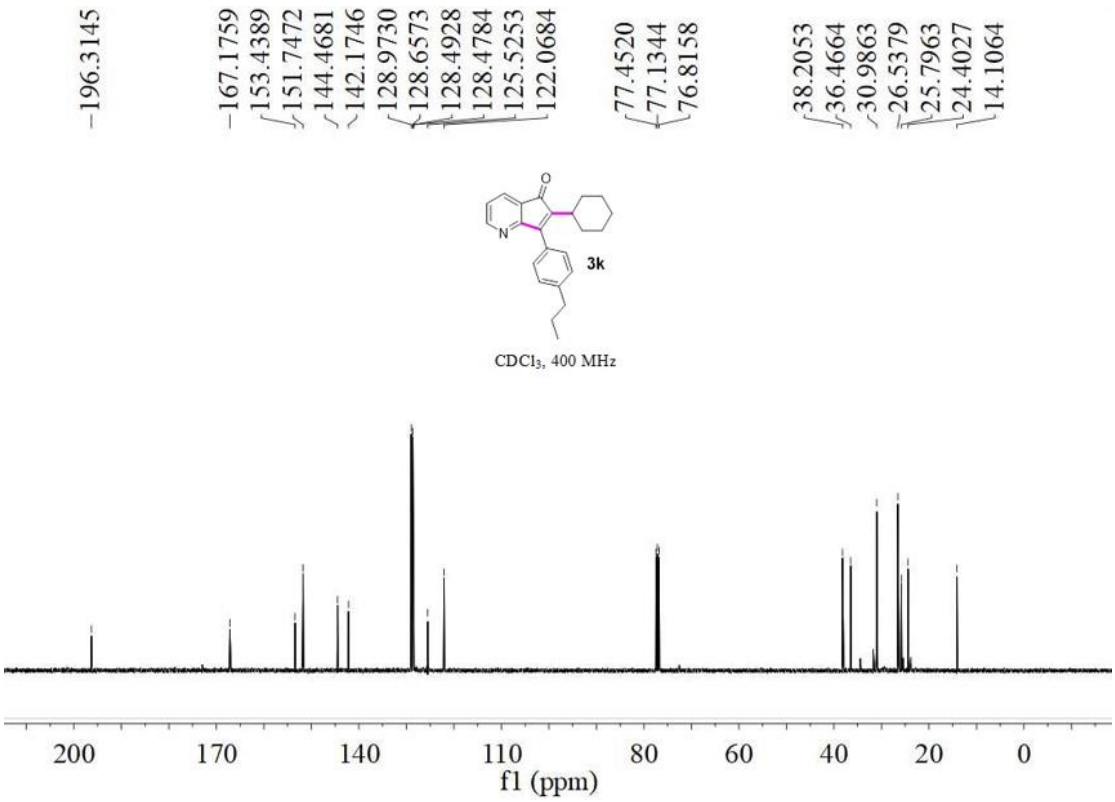

+TOF MS: 0.2557 to 0.2697 min from Sample 20 (XD-QUE-3-OME) of 20210105 (recalibrated06).wiff
a=7.018975754741454 90e-004, t0=-2.382239348146803 50e-001 (DuoSpray ())
Max. 2.4e6 cps.
320.1648
321.1700
Intensity, cps
m/z, Da
O
N
3i
OMe
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C18 H25 N2 O Cl | 320.1649 | -0.1928 | -0.6022 | 7
2 | C7 H24 N6 O8 | 320.165 | -0.2135 | -0.6669 | -1
3 | C21 H22 N O2 | 320.1645 | 0.2944 | 0.9195 | 11.5
4 | C2 H25 N10 O6 Cl | 320.1641 | 0.6419 | 2.0049 | -5
5 | C4 H27 N7 O7 Cl | 320.1655 | -0.7007 | -2.1887 | -5.5

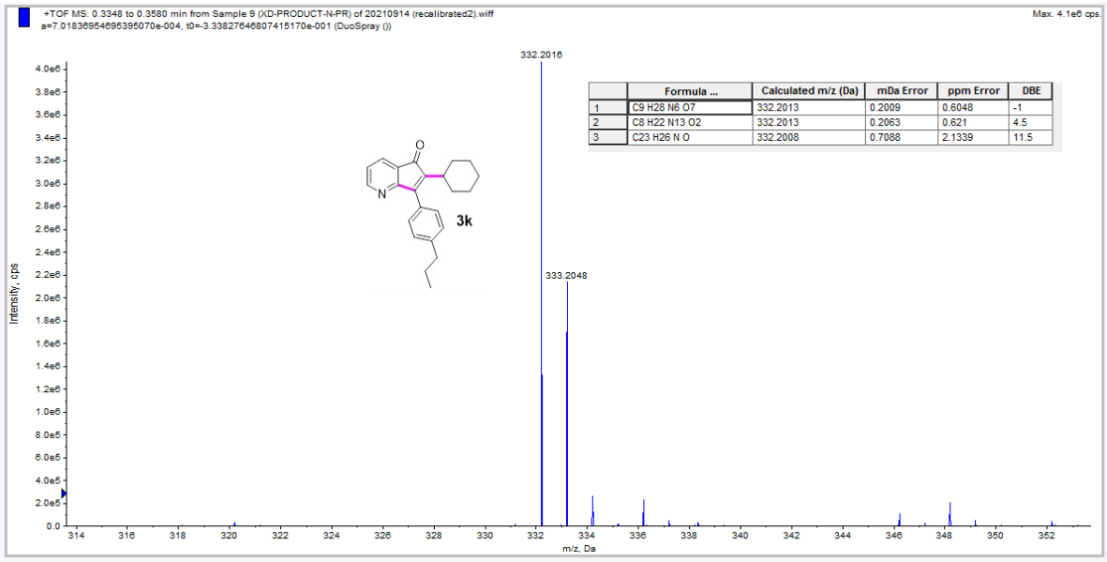

8.4266
8.4224
8.4135
8.4097
7.6329
7.6150
7.5836
7.5618
7.2687
7.0792
7.0715
7.0544
7.0502
3.8842
2.7016
2.6938
2.6716
2.6637
1.9715
1.9404
1.8108
1.7843
1.6541
1.6230
1.3135
1.2838
1.2585
1.2194
3j
OMe
CDCl3, 400 MHz
1.00
1.06
2.03
3.06
3.16
1.01
2.15
2.17
3.07
3.05
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

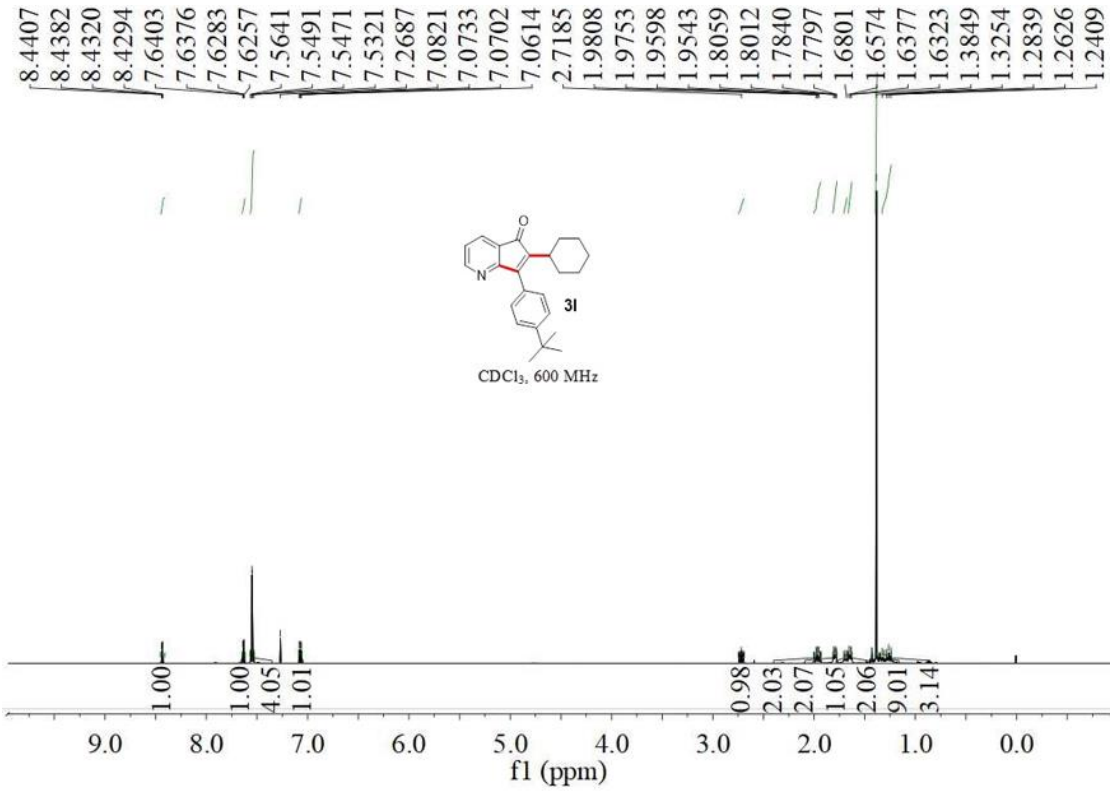

196.2510
167.1319
160.6845
153.0370
151.6393
141.5676
130.7396
128.3673
125.6419
123.5224
122.0632
114.0806
77.3256
77.1133
76.9009
55.4381
36.5492
30.9918
26.5927
25.8070
3j
OMe
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

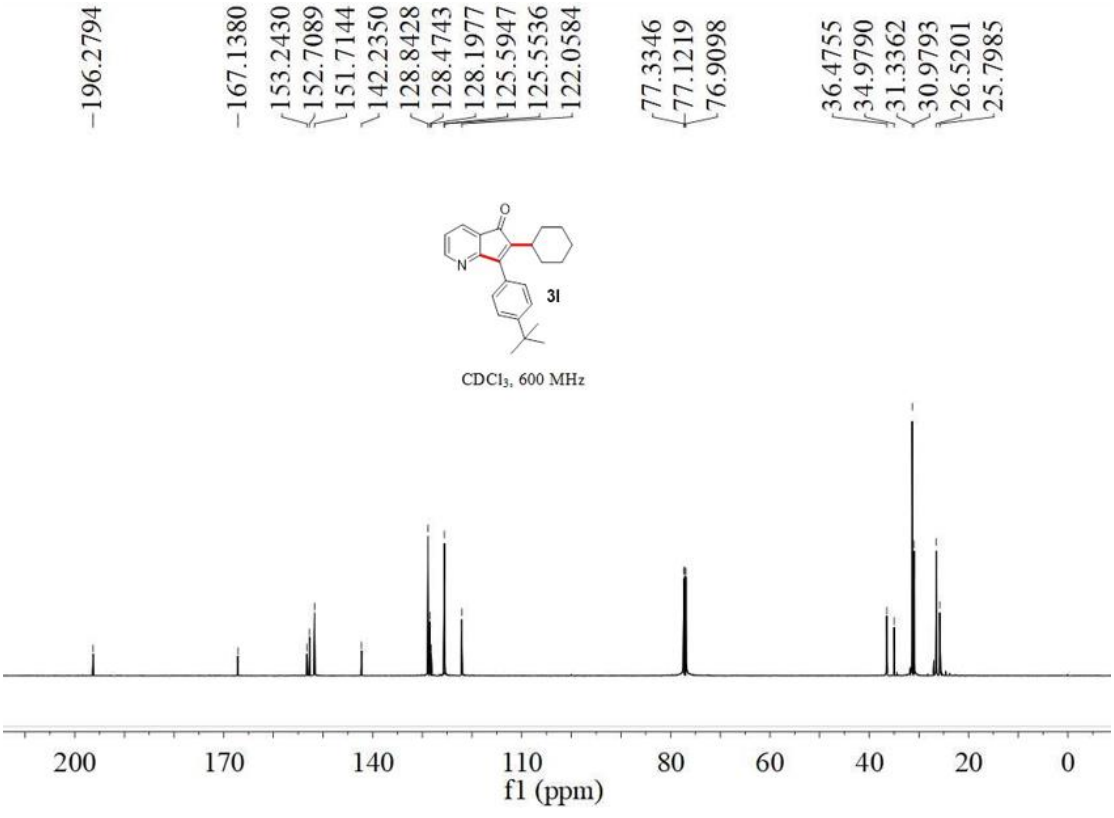

+TOF MS: 0.6970 to 0.7342 min from Sample 13 (XD-QUE-4-OME) of 20210105.wiff different calibrations (DuoSpray ())
Max. 4.0e6 cps
320.1646
321.1698
Intensity, cps
m/z, Da
3j
OMe
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C21 H22 N O2 | 320.1645 | 0.0944 | 0.2948 | 11.5
2 | C18 H25 N2 O Cl | 320.1649 | -0.3928 | -1.2268 | 7
3 | C7 H24 N6 O8 | 320.165 | -0.4135 | -1.2916 | -1
4 | C2 H25 N10 O6 Cl | 320.1641 | 0.4419 | 1.3803 | -5
5 | C4 H27 N7 O7 Cl | 320.1655 | -0.9007 | -2.8134 | -5.5
6 | C5 H22 N9 O7 | 320.1636 | 0.9291 | 2.902 | -0.5
7 | C16 H23 N5 Cl | 320.1636 | 0.9498 | 2.9668 | 7.5

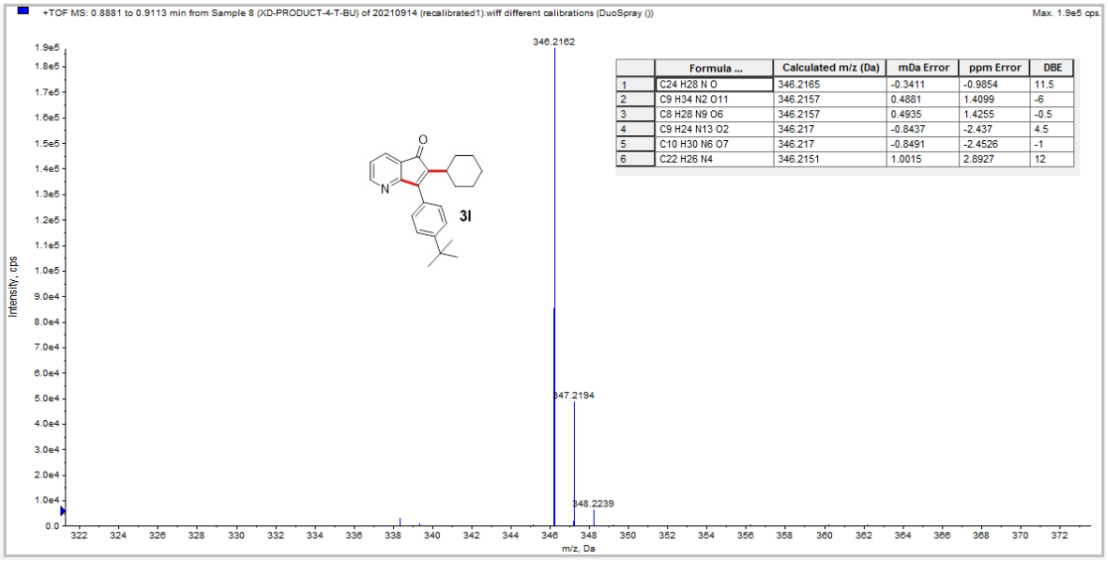

8.4282
8.4256
8.4230
8.4196
8.4169
8.4142
7.6334
7.6214
7.5100
7.4967
7.3492
7.3361
7.0768
7.0652
7.0561
2.6933
2.6787
2.6658
2.6530
1.9509
1.9303
1.7971
1.7758
1.7342
1.7213
1.7089
1.6965
1.6482
1.6261
1.2656
1.2444
1.2227
1.0203
1.0081
0.9959
3k
CDCl3, 600 MHz
1.00
1.02
2.02
2.09
1.03
3.04
2.06
2.04
3.05
2.13
3.08
3.00
10.5
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

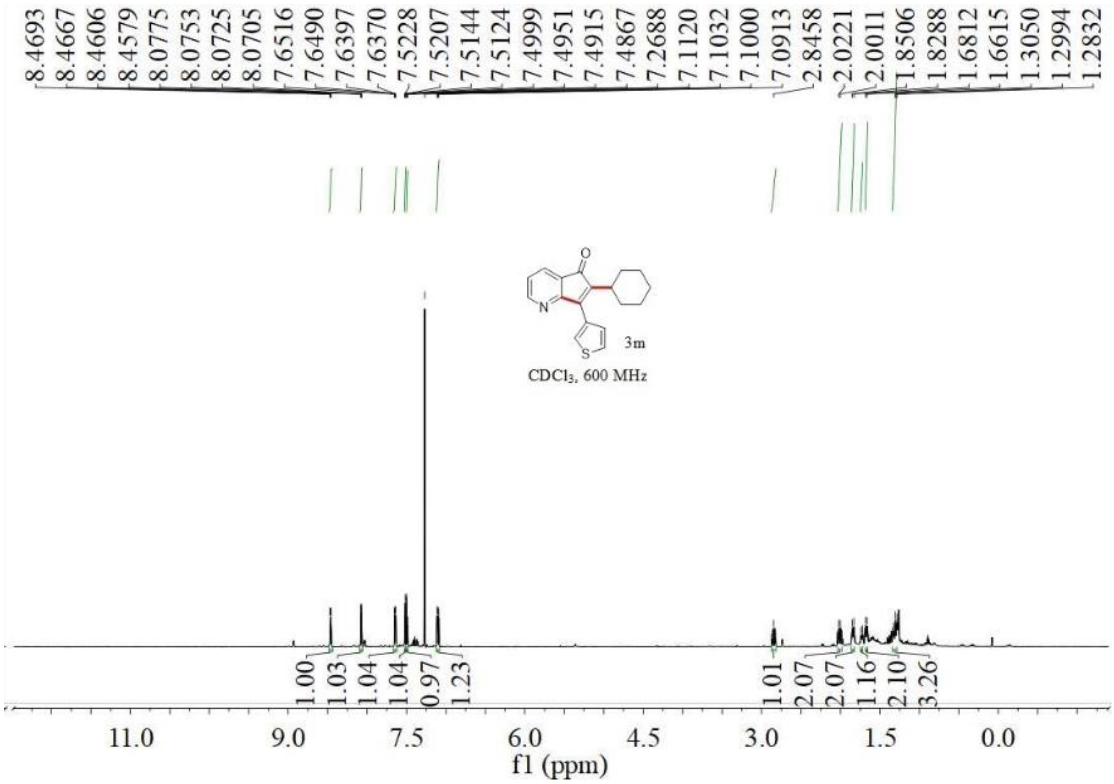

196.3145
167.1759
153.4389
151.7472
144.4681
142.1746
128.9730
128.6573
128.4928
128.4784
125.5253
122.0684
77.4520
77.1344
76.8158
38.2053
36.4664
30.9863
26.5379
25.7963
24.4027
14.1064
3k
CDCl3, 400 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

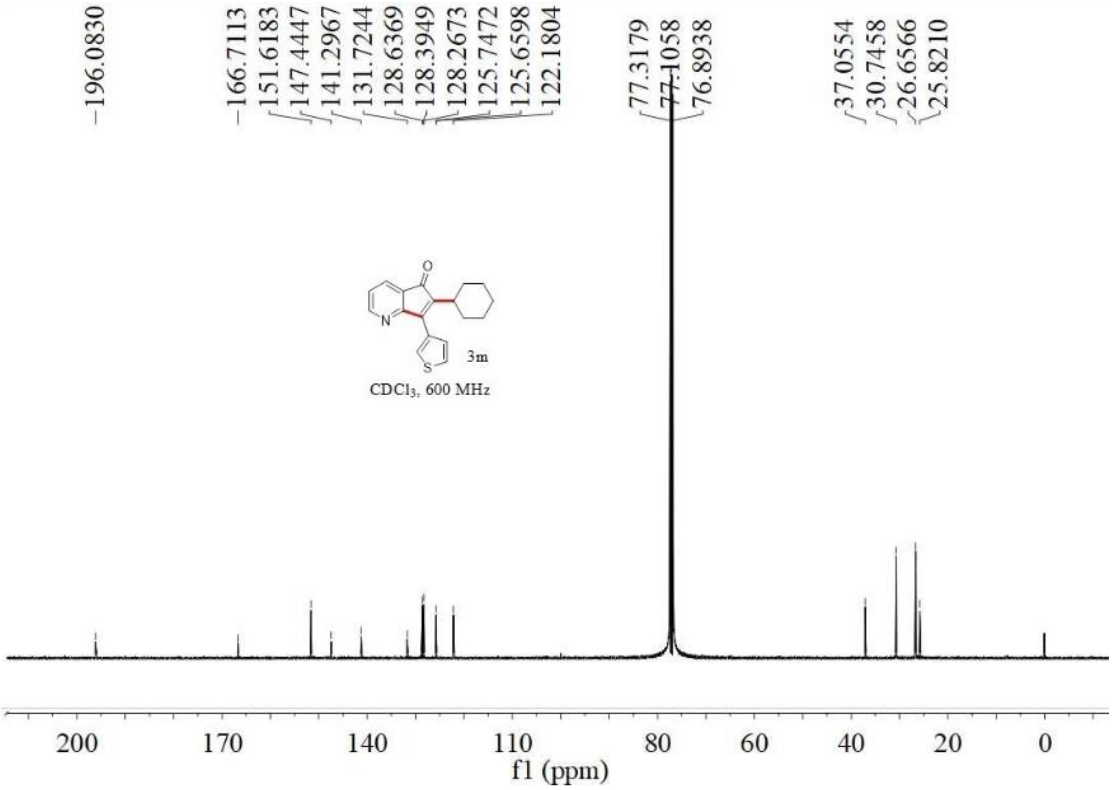

+TOF MS: 0.3348 to 0.3580 min from Sample 9 (XD-PRODUCT-N-PR) of 20210914 (recalibrated2).wiff
a=7.018369546963950 70e-004, t0=-3.338276468074151 70e-001 (DuoSpray ())
Max. 4.1e6 cps.
332.2016
333.2048
3k
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C9 H28 N6 O7 | 332.2013 | 0.2009 | 0.6048 | -1
2 | C8 H22 N13 O2 | 332.2013 | 0.2063 | 0.621 | 4.5
3 | C23 H26 N O | 332.2008 | 0.7088 | 2.1339 | 11.5
Intensity, cps
m/z, Da

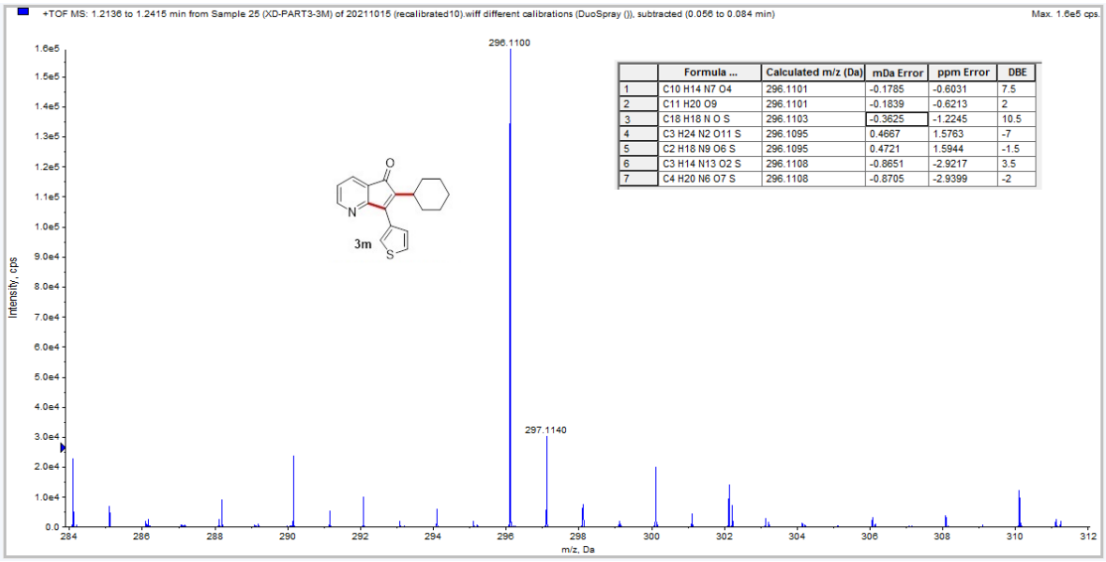

8.4407
8.4382
8.4320
8.4294
7.6403
7.6376
7.6283
7.6257
7.5641
7.5491
7.5471
7.5321
7.2687
7.0821
7.0733
7.0702
7.0614
2.7185
1.9808
1.9753
1.9598
1.9543
1.8059
1.8012
1.7840
1.7797
1.6801
1.6574
1.6377
1.6323
1.3849
1.3254
1.2839
1.2626
1.2409
3l
CDCl3, 600 MHz
1.00
1.00
4.05
1.01
0.98
2.03
2.07
1.05
2.06
9.01
3.14
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


196.2794
167.1380
153.2430
152.7089
151.7144
142.2350
128.8428
128.4743
128.1977
125.5947
125.5536
122.0584
77.3346
77.1219
76.9098
36.4755
34.9790
31.3362
30.9793
26.5201
25.7985
3l
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

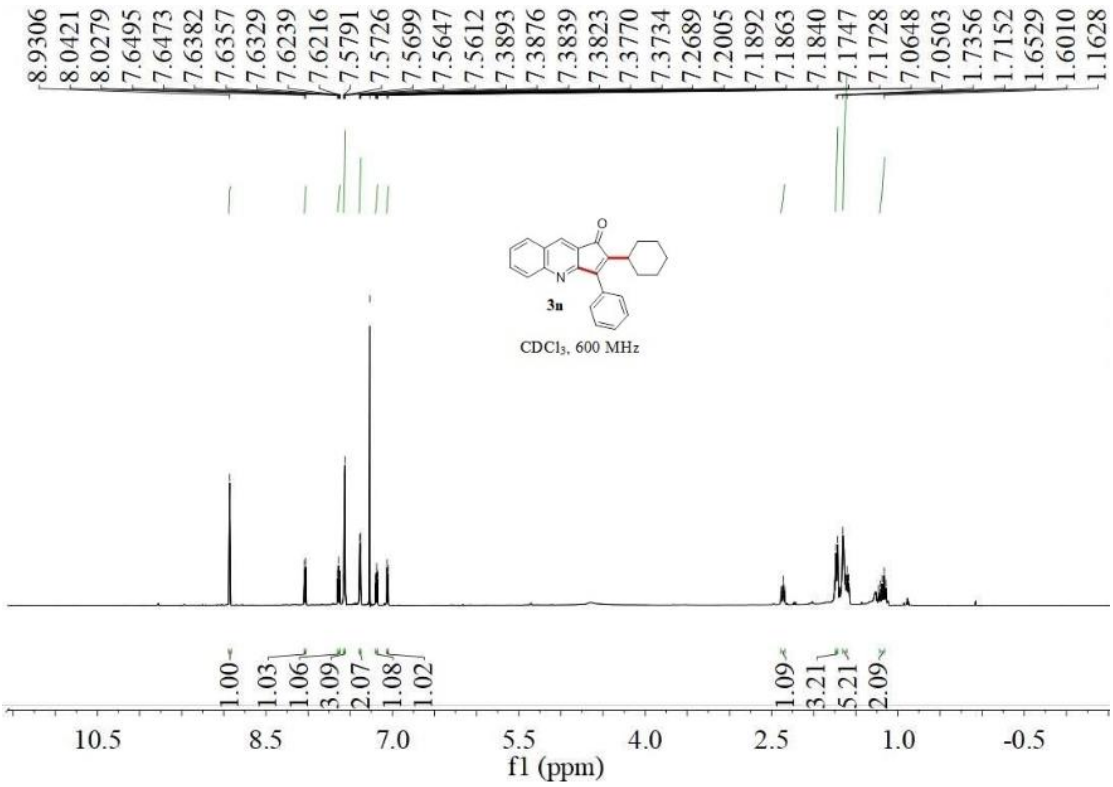

+TOF MS: 0.8881 to 0.9113 min from Sample 8 (XD-PRODUCT-4-T-BU) of 20210914 (recalibrated1).wiff different calibrations (DuoSpray ())
Max. 1.9e5 cps.
346.2162
347.2194
348.2239
Intensity, cps
m/z, Da
3l
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C24 H28 N O | 346.2165 | -0.3411 | -0.9854 | 11.5
2 | C9 H34 N2 O11 | 346.2157 | 0.4881 | 1.4099 | -6
3 | C8 H28 N9 O6 | 346.2157 | 0.4935 | 1.4255 | -0.5
4 | C9 H24 N13 O2 | 346.217 | -0.8437 | -2.437 | 4.5
5 | C10 H30 N6 O7 | 346.217 | -0.8491 | -2.4526 | -1
6 | C22 H26 N4 | 346.2151 | 1.0015 | 2.8927 | 12

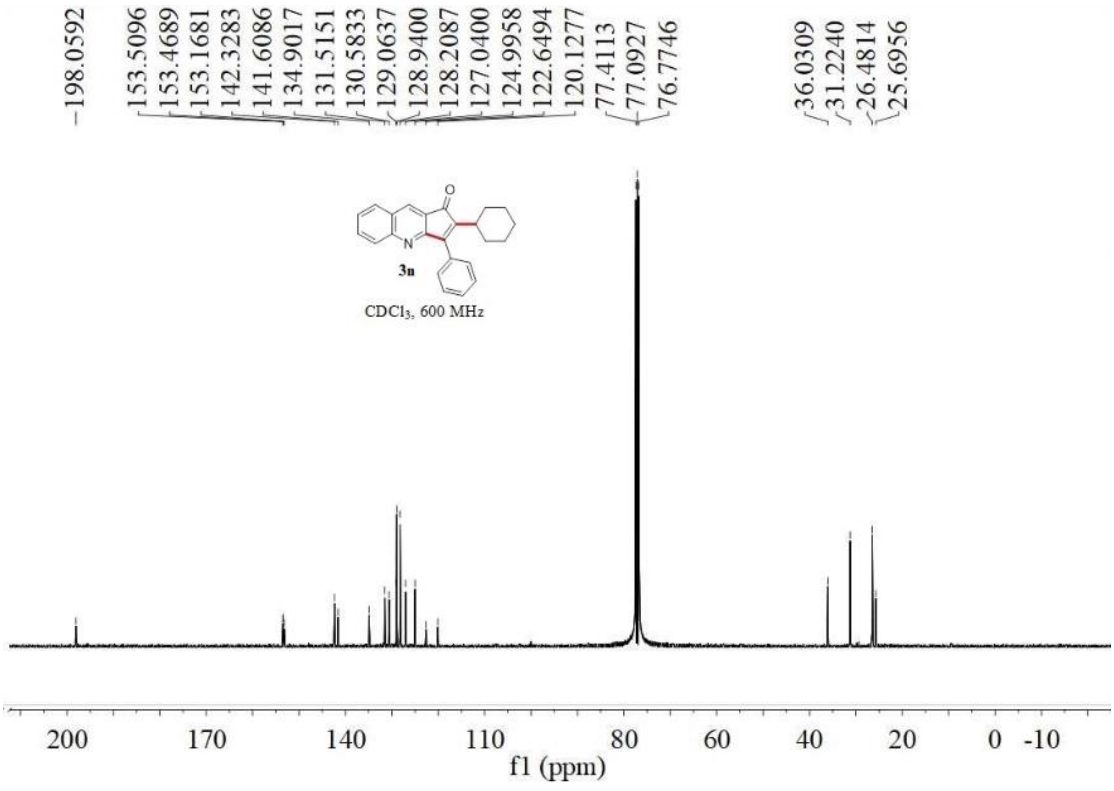

8.4693
8.4667
8.4606
8.4579
8.0775
8.0753
8.0725
8.0705
7.6516
7.6490
7.6397
7.6370
7.5228
7.5207
7.5144
7.5124
7.4999
7.4951
7.4915
7.4867
7.2688
7.1120
7.1032
7.1000
7.0913
2.8458
2.0221
2.0011
1.8506
1.8288
1.6812
1.6615
1.3050
1.2994
1.2832
3m
CDCl3, 600 MHz
1.00
1.03
1.04
1.04
0.97
1.23
1.01
2.07
2.07
1.16
2.10
3.26
11.0
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)

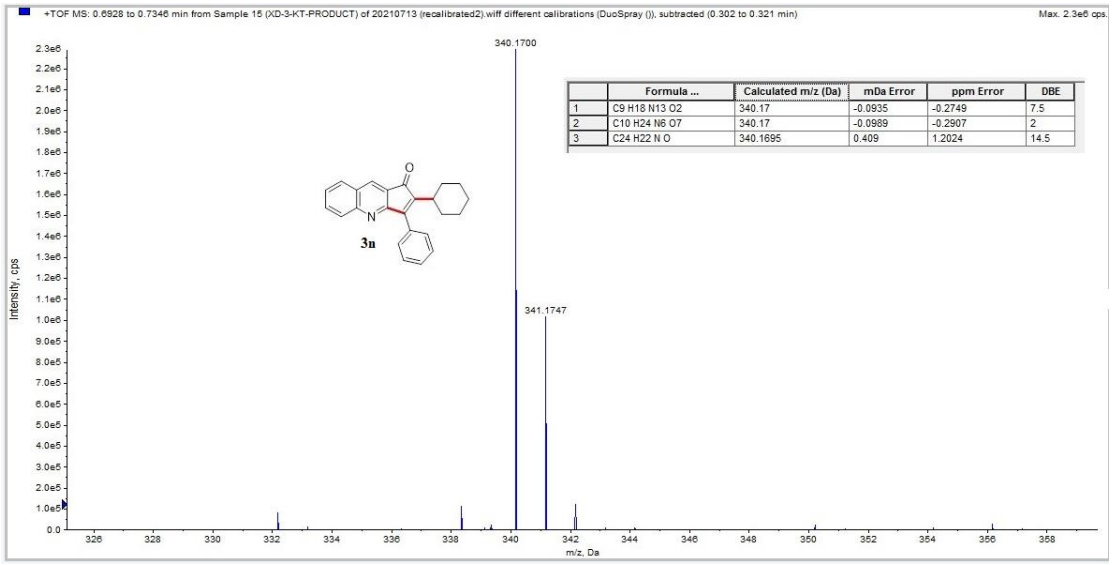




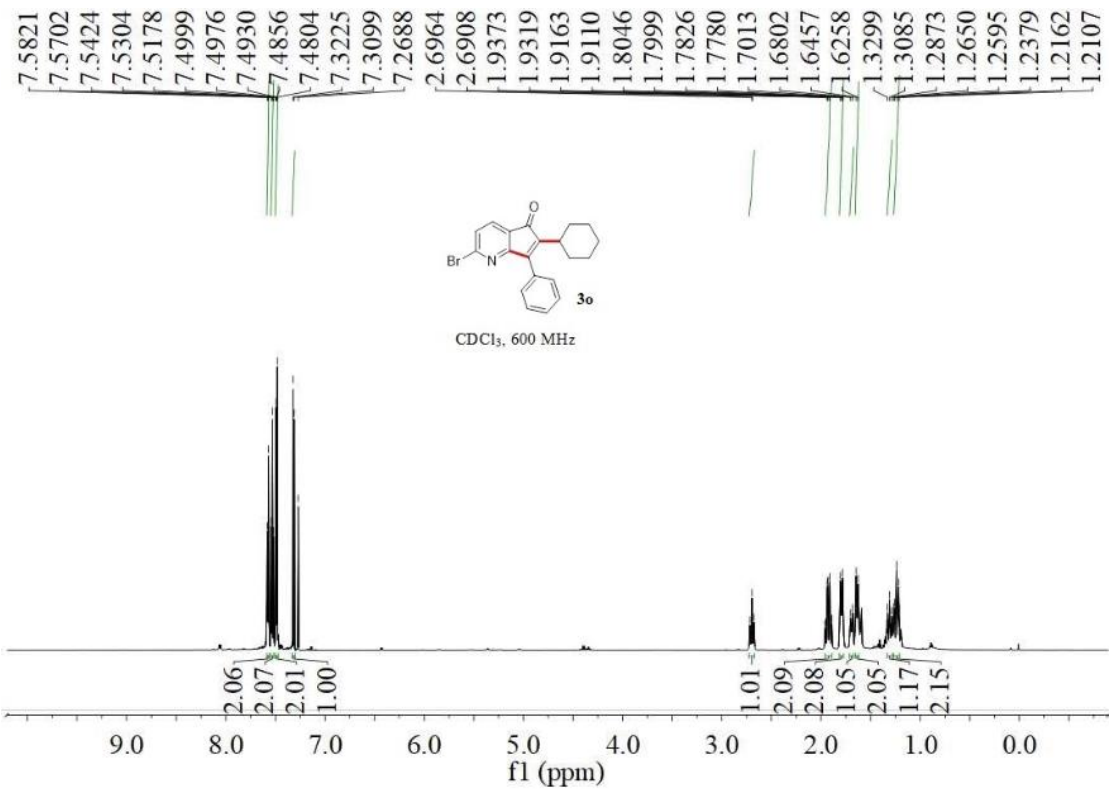
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C10 H14 N7 O4	296.1101	-0.1785	-0.6031	7.5
2	C11 H20 O9	296.1101	-0.1839	-0.6213	2
3	C18 H18 N O S	296.1103	-0.3625	-1.2245	10.5
4	C3 H24 N2 O11 S	296.1095	0.4667	1.5763	-7
5	C2 H18 N9 O6 S	296.1095	0.4721	1.5944	-1.5
6	C3 H14 N13 O2 S	296.1108	-0.8651	-2.9217	3.5
7	C4 H20 N6 O7 S	296.1108	-0.8705	-2.9399	-2

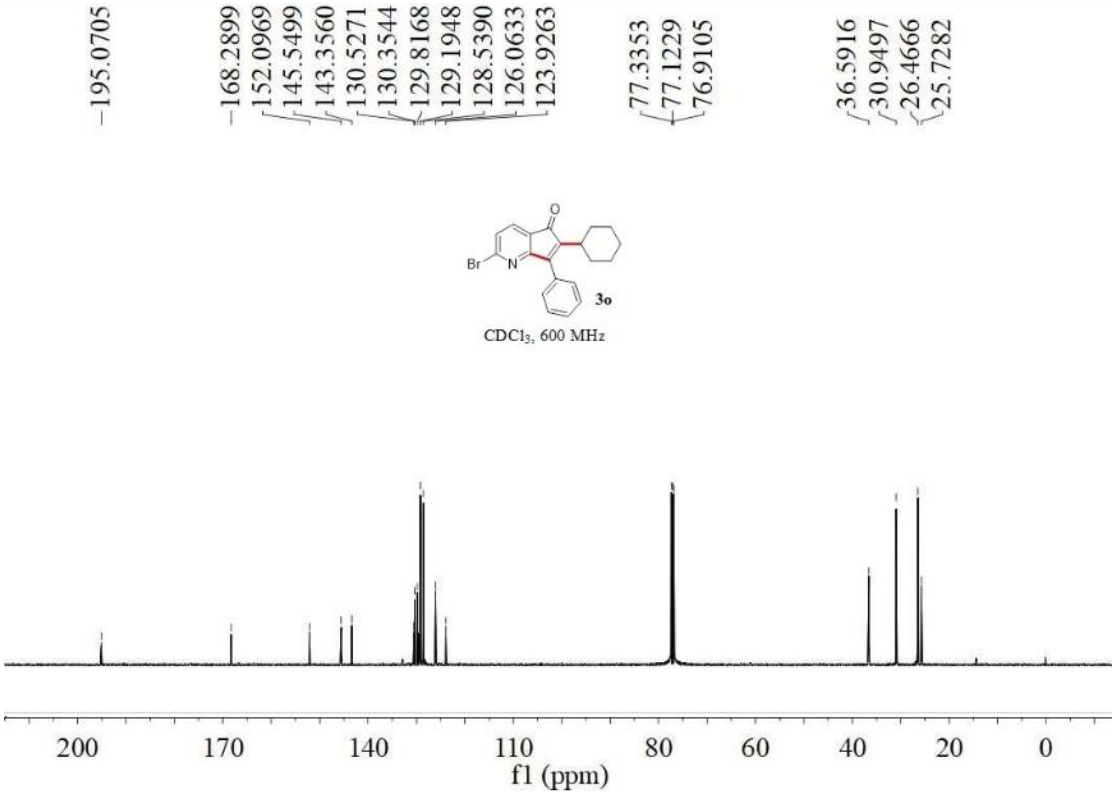

8.9306
8.0421
8.0279
7.6495
7.6473
7.6382
7.6357
7.6329
7.6239
7.6216
7.5791
7.5726
7.5699
7.5647
7.5612
7.3893
7.3876
7.3839
7.3823
7.3770
7.3734
7.2689
7.2005
7.1892
7.1863
7.1840
7.1747
7.1728
7.0648
7.0503
1.7356
1.7152
1.6529
1.6010
1.1628
3n
CDCl3, 600 MHz
1.00
1.03
1.06
3.09
2.07
1.08
1.02
1.09
3.21
5.21
2.09
10.5
8.5
7.0
5.5
4.0
2.5
1.0
-0.5
f1 (ppm)

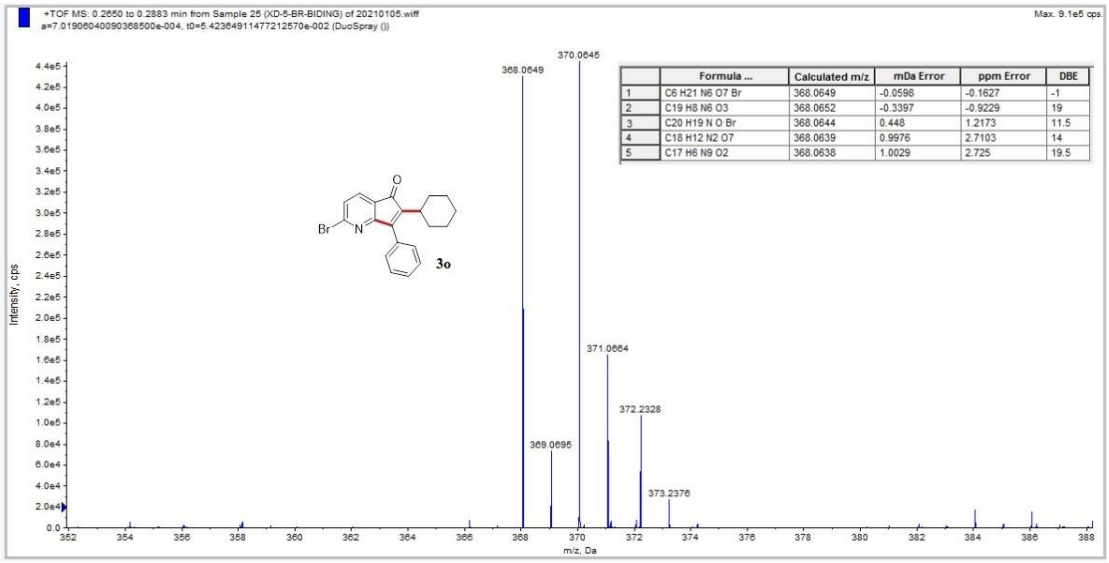

198.0592
153.5096
153.4689
153.1681
142.3283
141.6086
134.9017
131.5151
130.5833
129.0637
128.9400
128.2087
127.0400
124.9958
122.6494
120.1277
77.4113
77.0927
76.7746
36.0309
31.2240
26.4814
25.6956
3n
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
-10
f1 (ppm)

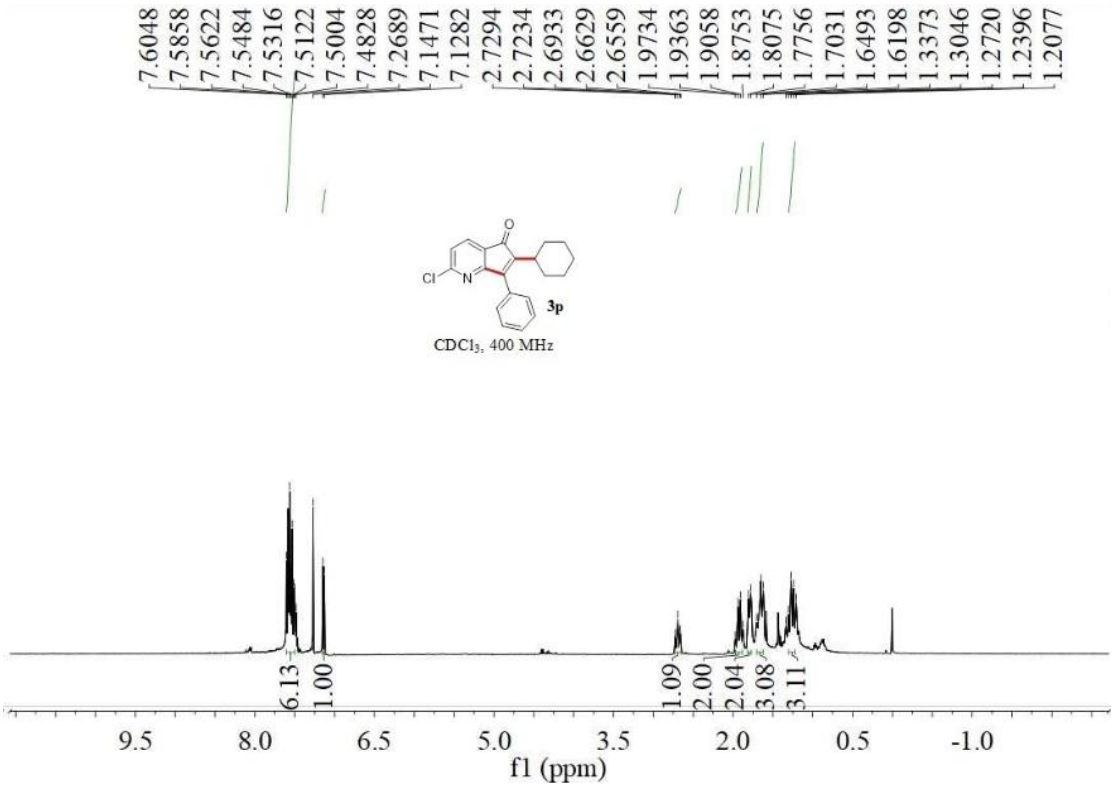


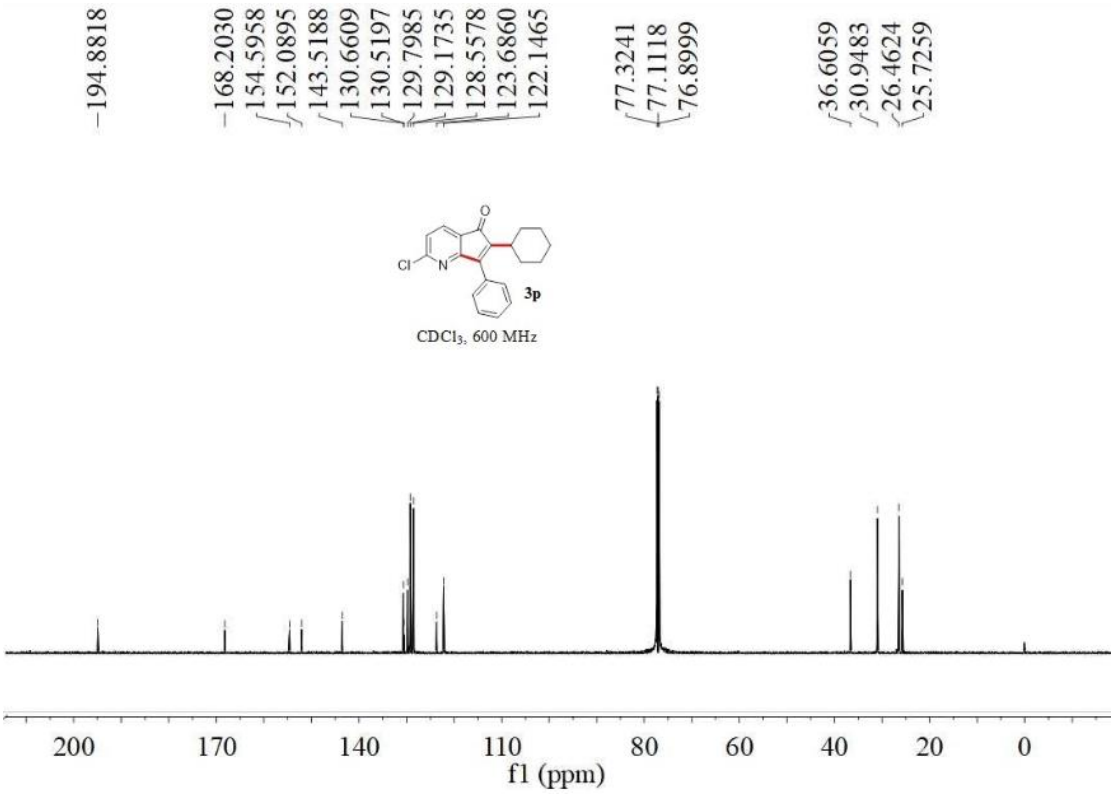
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C9 H18 N13 O2	340.17	-0.0935	-0.2749	7.5
2	C10 H24 N6 O7	340.17	-0.0989	-0.2907	2
3	C24 H22 N O	340.1695	0.409	1.2024	14.5

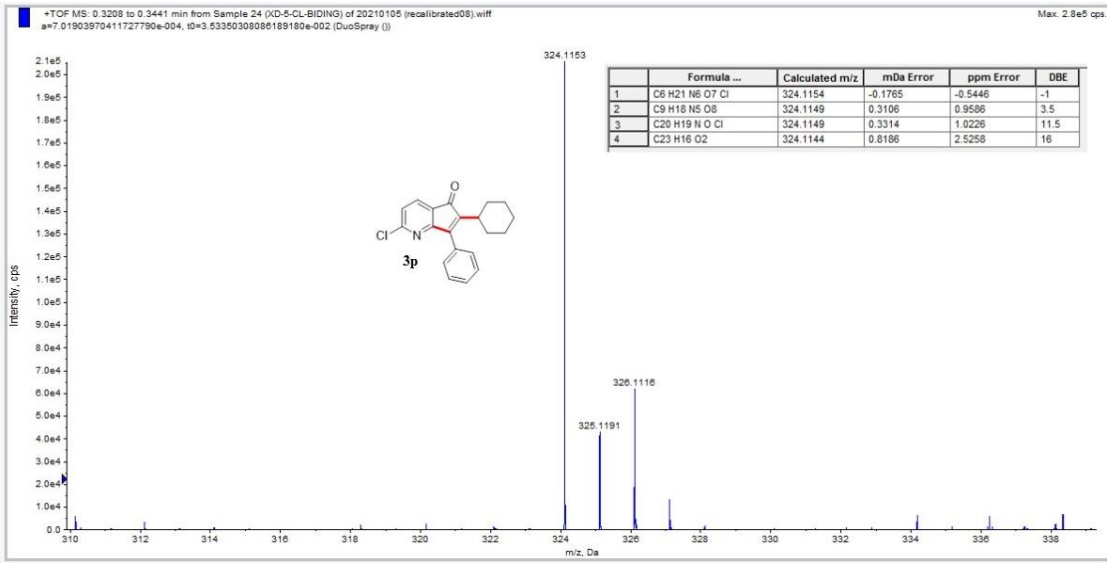



195.0705
168.2899
152.0969
145.5499
143.3560
130.5271
130.3544
129.8168
129.1948
128.5390
126.0633
123.9263
77.3353
77.1229
76.9105
36.5916
30.9497
26.4666
25.7282
Br
N
O
3o
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

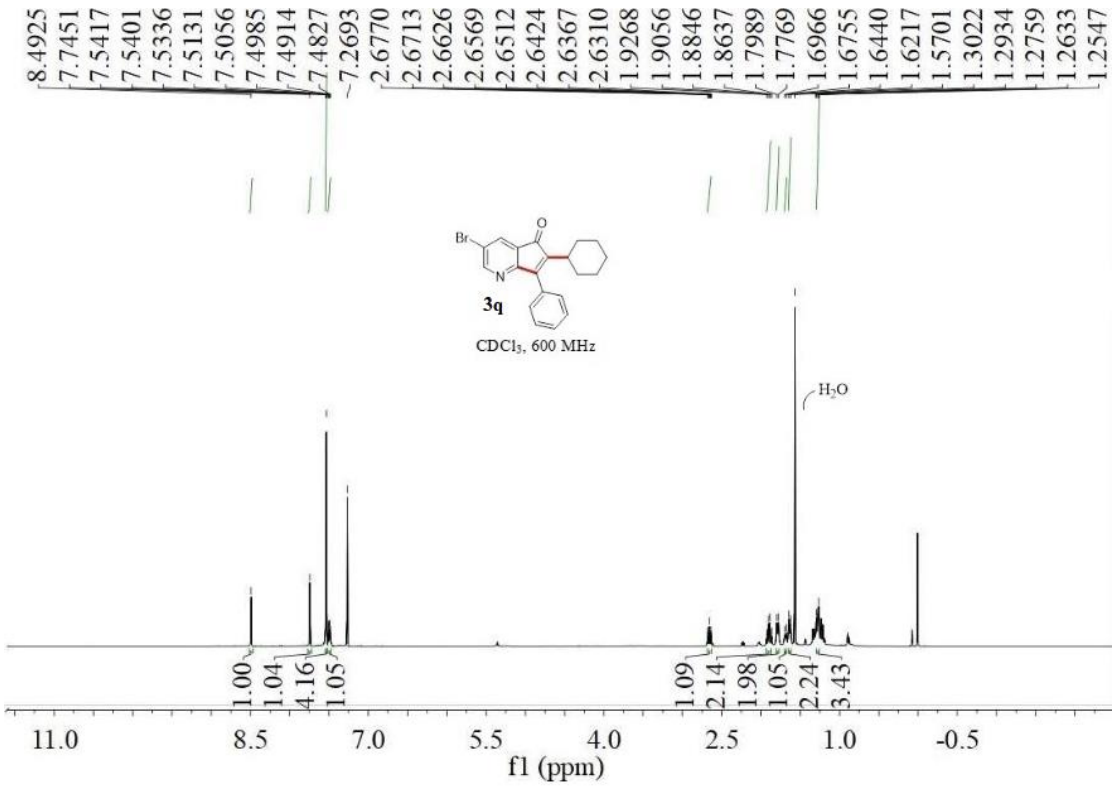

+TOF MS: 0.2650 to 0.2883 min from Sample 25 (XD-5-BR-BIDING) of 20210105.wiff
Max. 9.1e5 cps.
370.0645
368.0649
371.0664
372.2328
369.0695
373.2376
Intensity, cps
m/z, Da
Br
N
O
3o
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C6 H21 N6 O7 Br | 368.0649 | -0.0598 | -0.1627 | -1
2 | C19 H8 N6 O3 | 368.0652 | -0.3397 | -0.9229 | 19
3 | C20 H19 N O Br | 368.0644 | 0.448 | 1.2173 | 11.5
4 | C18 H12 N2 O7 | 368.0639 | 0.9976 | 2.7103 | 14
5 | C17 H6 N9 O2 | 368.0638 | 1.0029 | 2.725 | 19.5

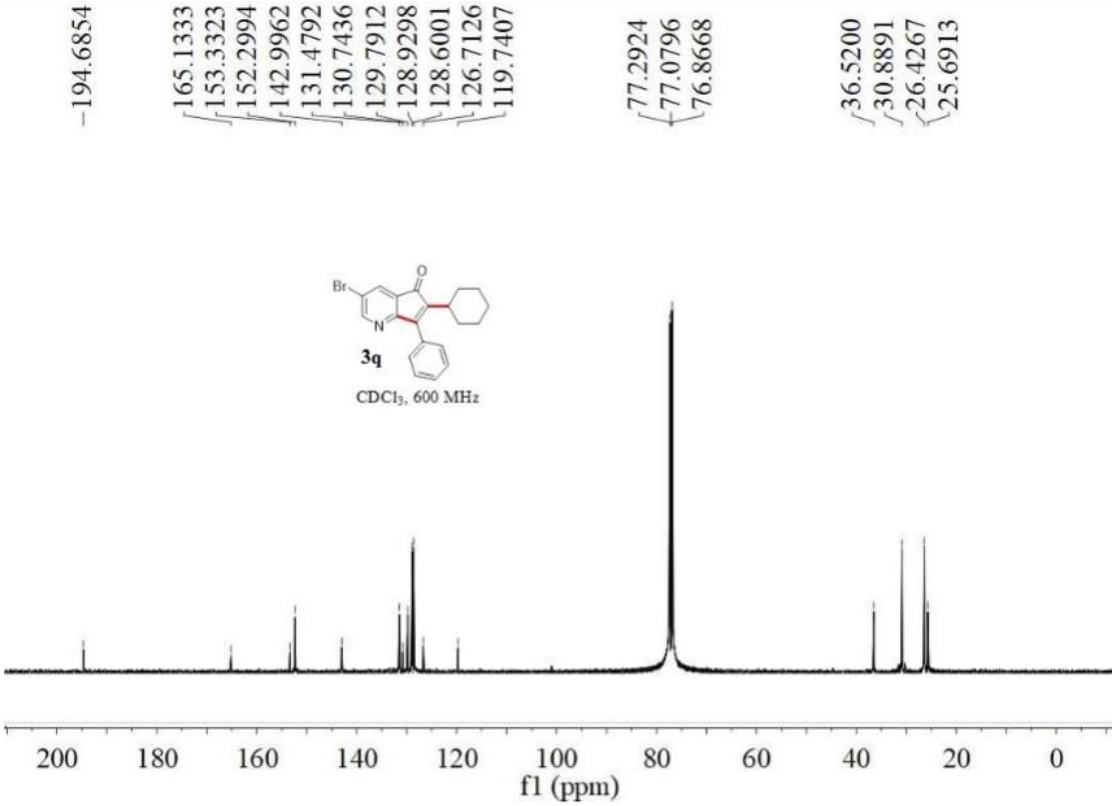

7.6048
7.5858
7.5622
7.5484
7.5316
7.5122
7.5004
7.4828
7.2689
7.1471
7.1282
2.7294
2.7234
2.6933
2.6629
2.6559
1.9734
1.9363
1.9058
1.8753
1.8075
1.7756
1.7031
1.6493
1.6198
1.3373
1.3046
1.2720
1.2396
1.2077
O
Cl
N
3p
CDCl3, 400 MHz
6.13
1.00
1.09
2.00
2.04
3.08
3.11
9.5
8.0
6.5
5.0
3.5
2.0
0.5
-1.0
f1 (ppm)

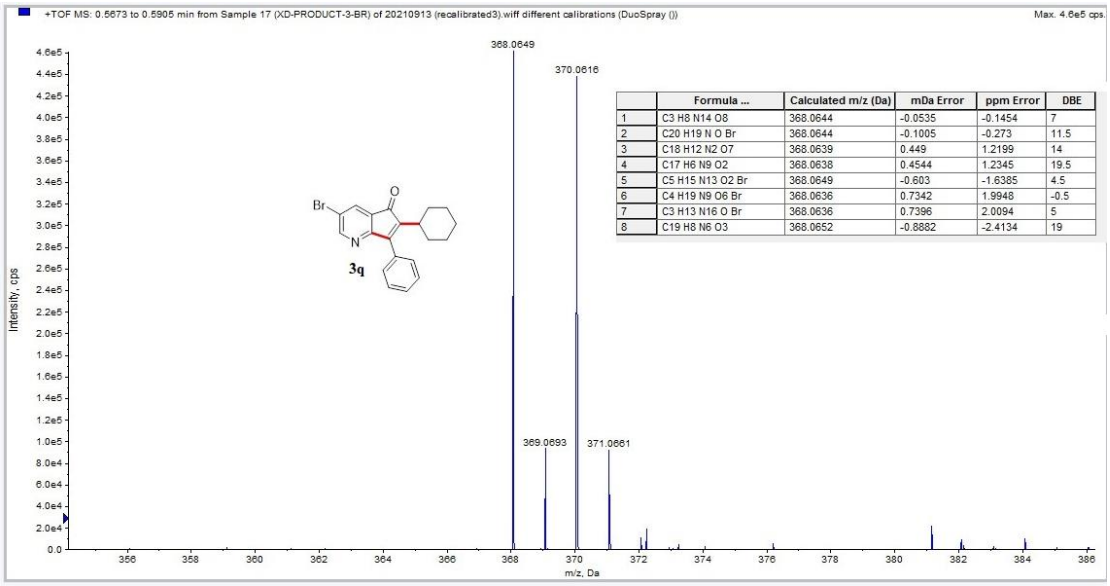

194.8818
168.2030
154.5958
152.0895
143.5188
130.6609
130.5197
129.7985
129.1735
128.5578
123.6860
122.1465
77.3241
77.1118
76.8999
36.6059
30.9483
26.4624
25.7259
O
Cl
N
3p
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)



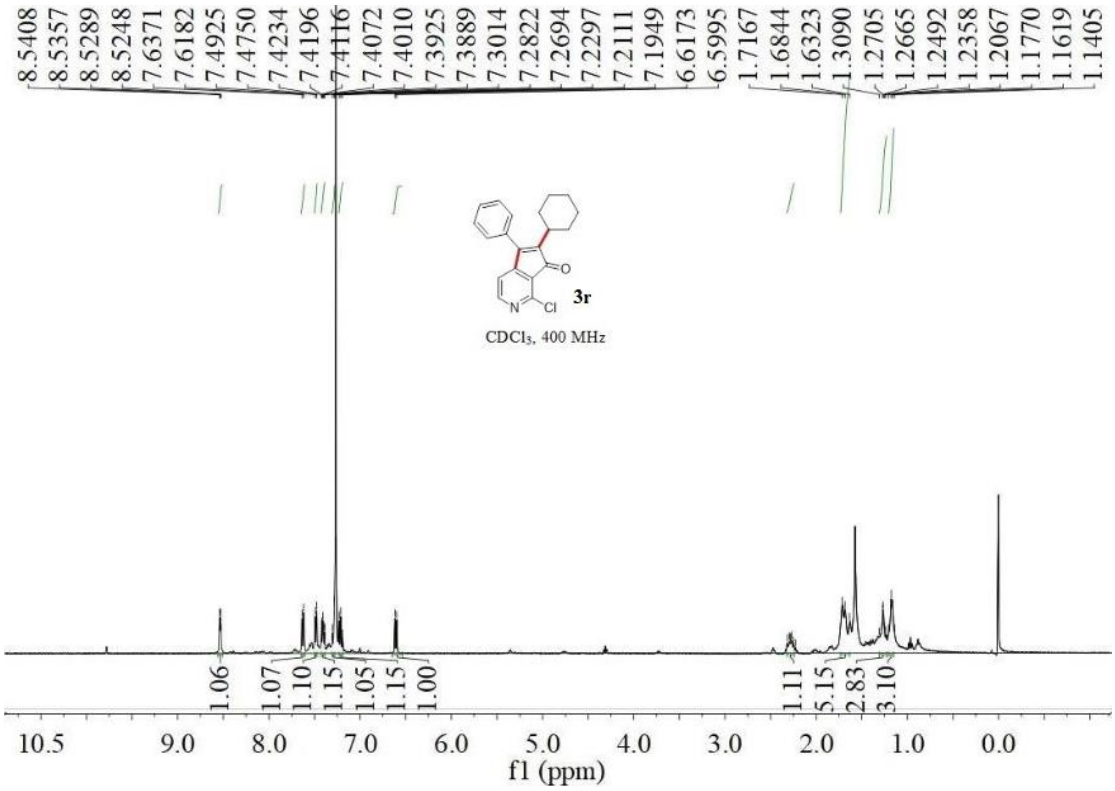
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C6 H21 N6 O7 Cl	324.1154	-0.1765	-0.5446	-1
2	C9 H18 N5 O8	324.1149	0.3106	0.9586	3.5
3	C20 H19 N O Cl	324.1149	0.3314	1.0226	11.5
4	C23 H16 O2	324.1144	0.8186	2.5258	16

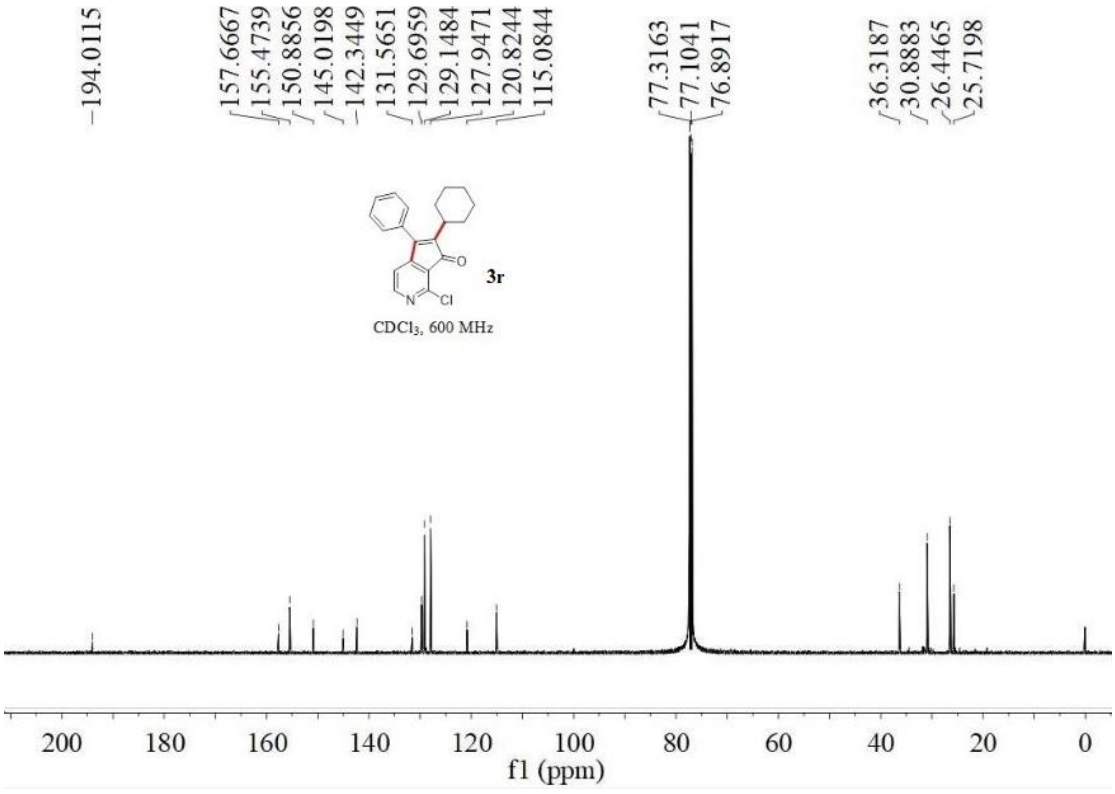


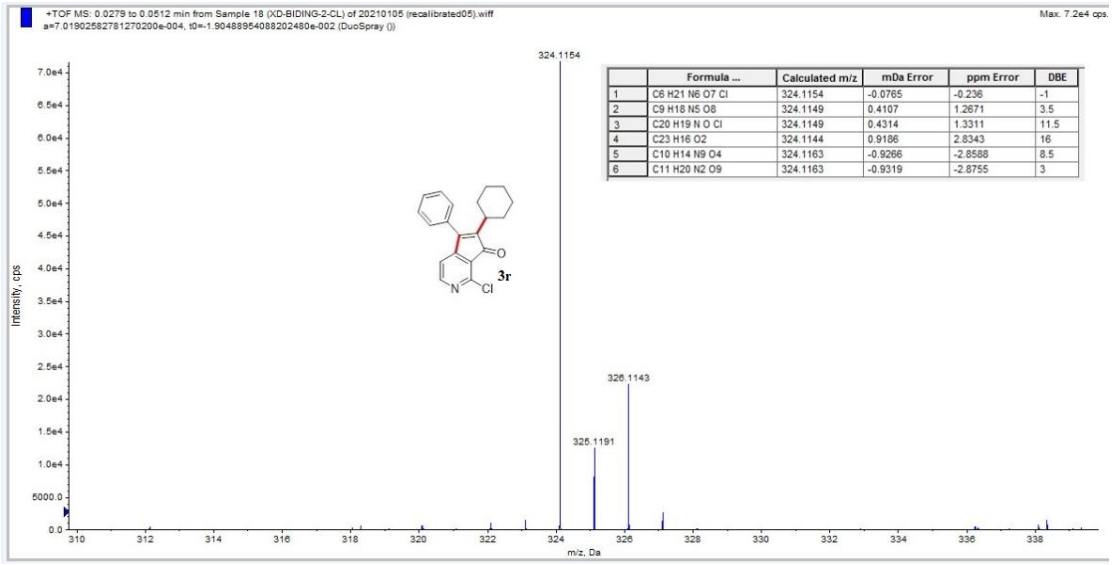




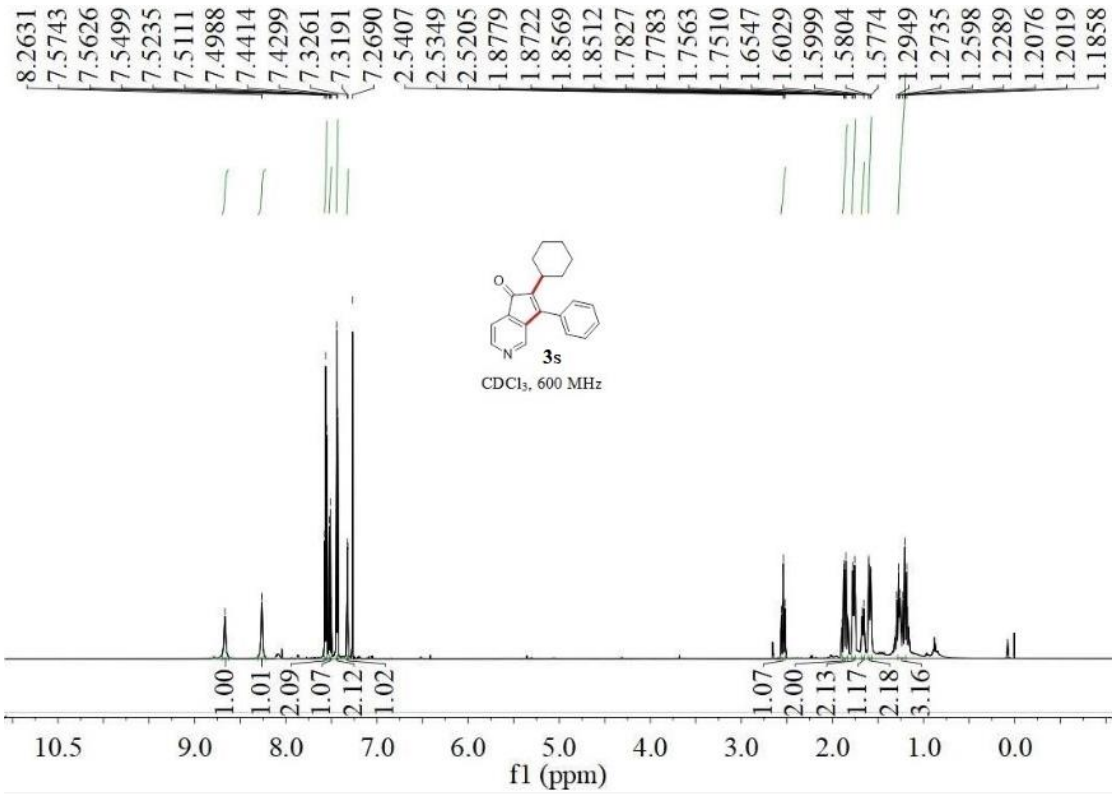
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C3 H8 N14 O8	368.0644	-0.0535	-0.1454	7
2	C20 H19 N O Br	368.0644	-0.1005	-0.273	11.5
3	C18 H12 N2 O7	368.0639	0.449	1.2199	14
4	C17 H6 N9 O2	368.0638	0.4544	1.2345	19.5
5	C5 H15 N13 O2 Br	368.0649	-0.603	-1.6385	4.5
6	C4 H19 N9 O6 Br	368.0636	0.7342	1.9948	-0.5
7	C3 H13 N16 O Br	368.0636	0.7396	2.0094	5
8	C19 H8 N6 O3	368.0652	-0.8882	-2.4134	19

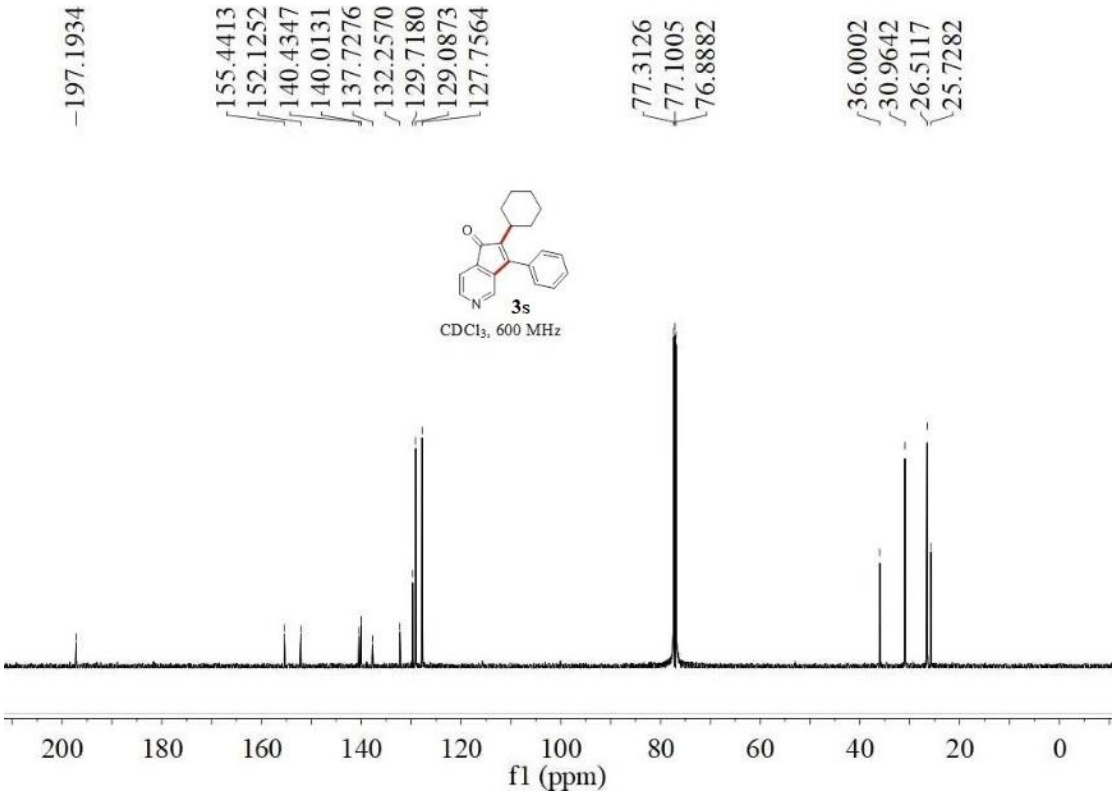

8.5408
8.5357
8.5289
8.5248
7.6371
7.6182
7.4925
7.4750
7.4234
7.4196
7.4116
7.4072
7.4010
7.3925
7.3889
7.3014
7.2822
7.2694
7.2297
7.2111
7.1949
6.6173
6.5995
1.7167
1.6844
1.6323
1.3090
1.2705
1.2665
1.2492
1.2358
1.2067
1.1770
1.1619
1.1405
3r
CDCl3, 400 MHz
1.06
1.07
1.10
1.15
1.05
1.15
1.00
1.11
5.15
2.83
3.10
10.5
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

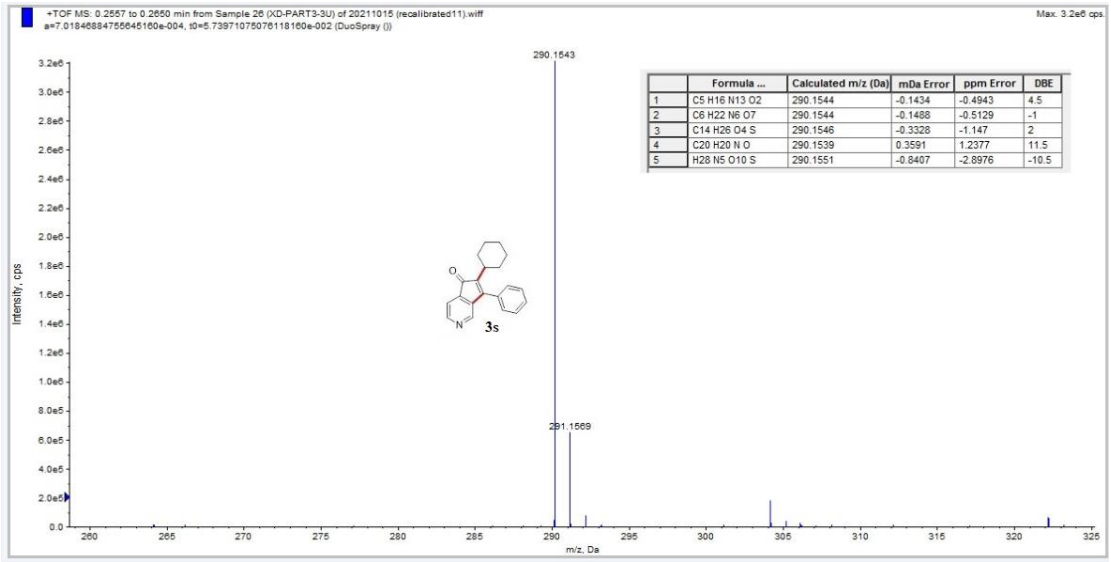

194.0115
157.6667
155.4739
150.8856
145.0198
142.3449
131.5651
129.6959
129.1484
127.9471
120.8244
115.0844
77.3163
77.1041
76.8917
36.3187
30.8883
26.4465
25.7198
3r
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

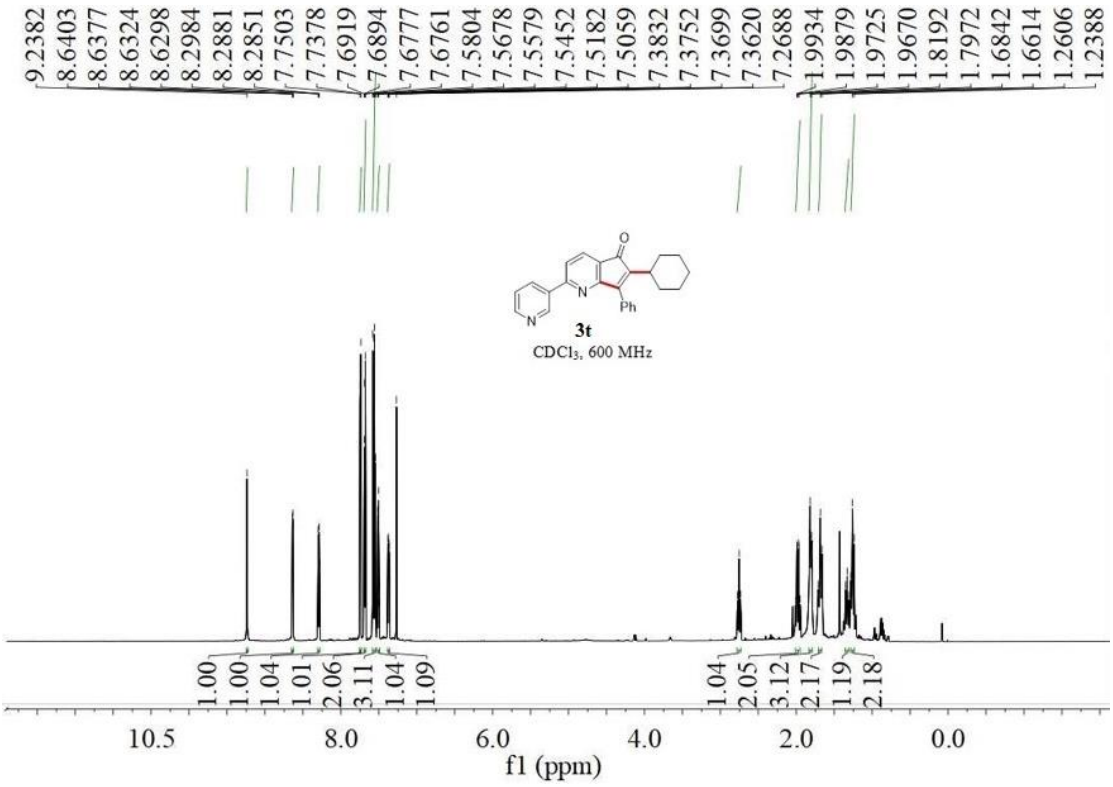


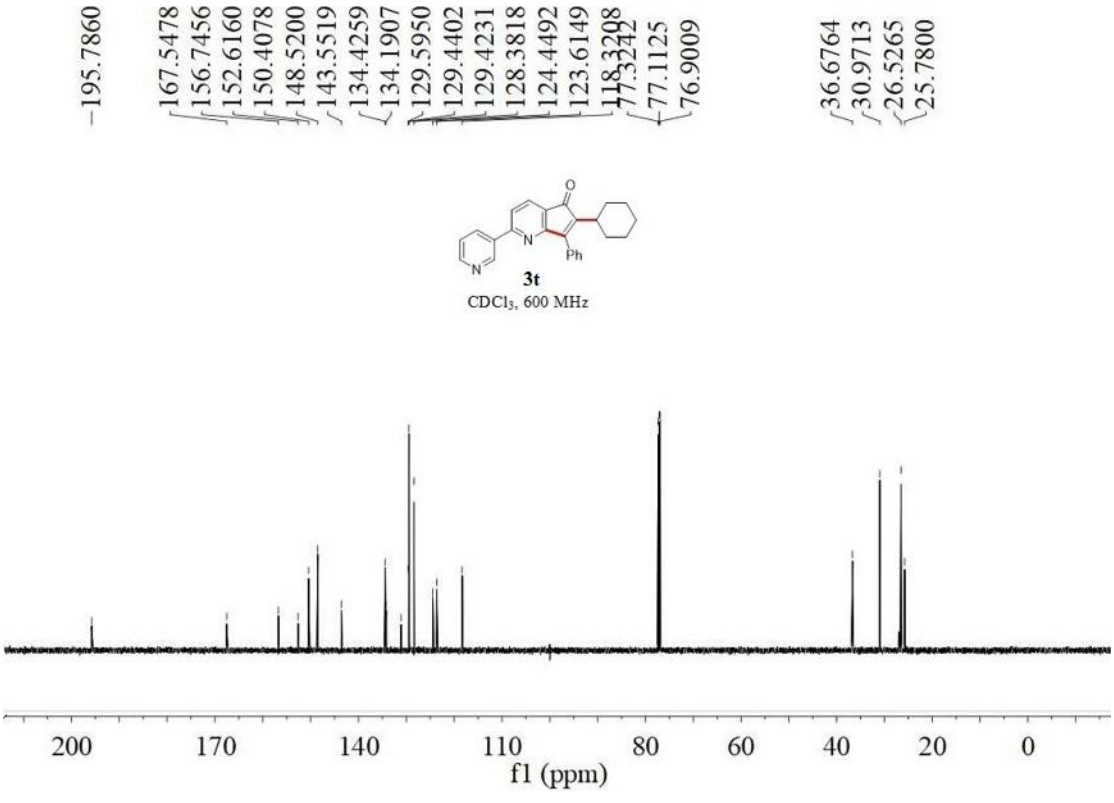
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C6 H21 N6 O7 Cl	324.1154	-0.0765	-0.236	-1
2	C9 H18 N5 O8	324.1149	0.4107	1.2671	3.5
3	C20 H19 N O Cl	324.1149	0.4314	1.3311	11.5
4	C23 H16 O2	324.1144	0.9186	2.8343	16
5	C10 H14 N9 O4	324.1163	-0.9266	-2.8588	8.5
6	C11 H20 N2 O9	324.1163	-0.9319	-2.8755	3

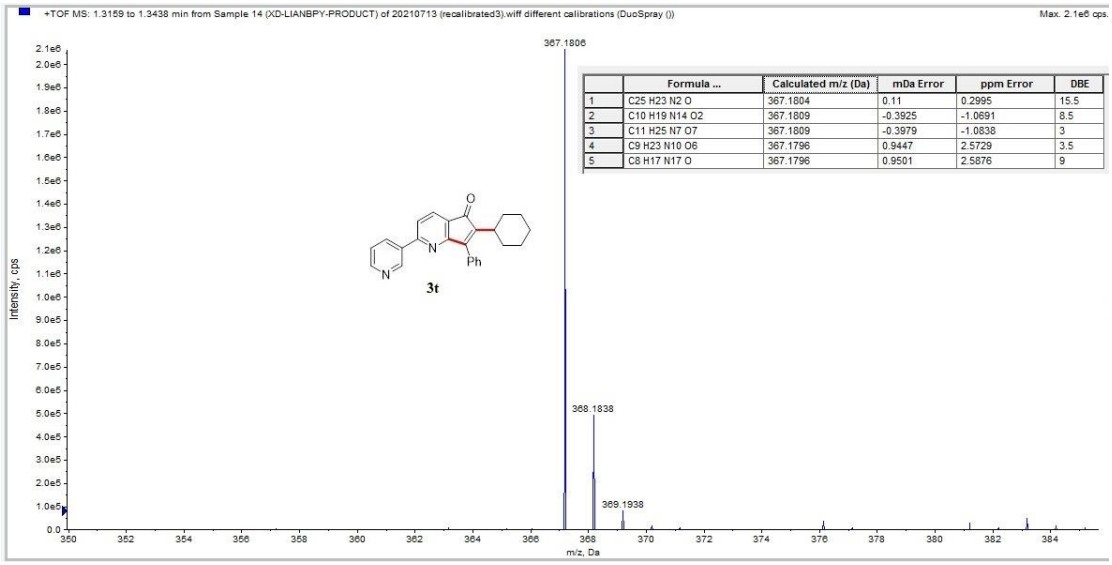



197.1934
155.4413
152.1252
140.4347
140.0131
137.7276
132.2570
129.7180
129.0873
127.7564
77.3126
77.1005
76.8882
36.0002
30.9642
26.5117
25.7282
3s
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

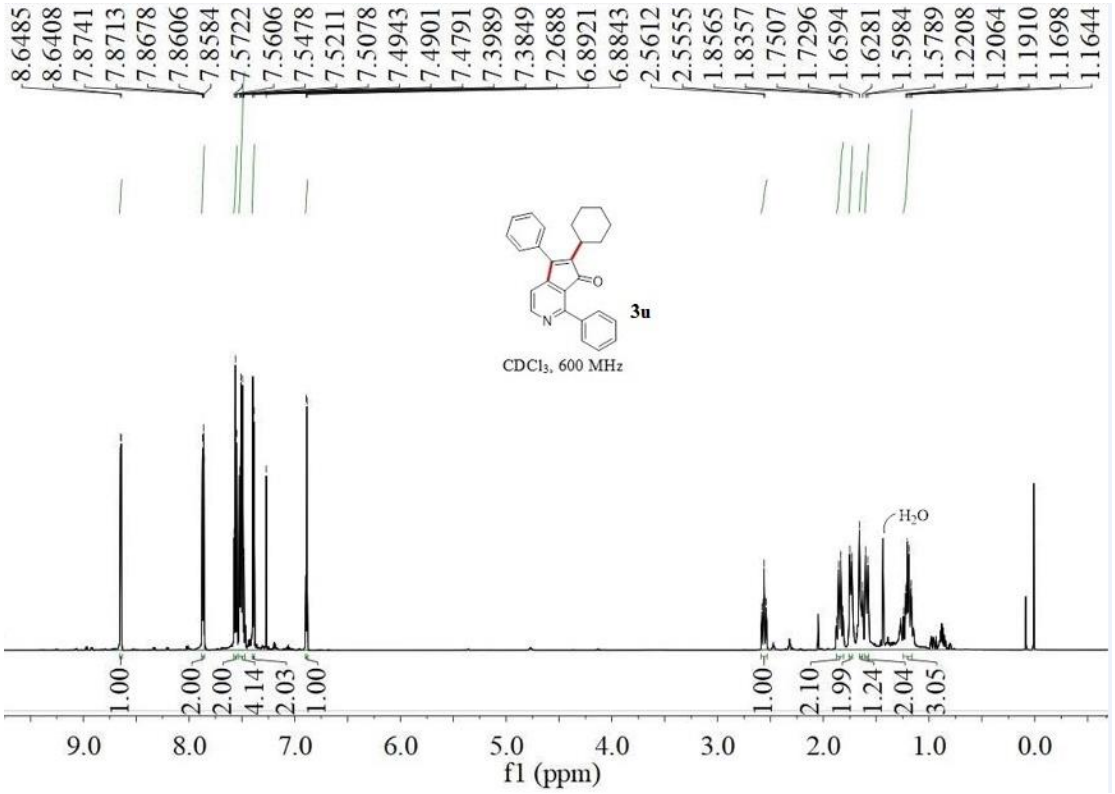

+TOF MS: 0.2557 to 0.2650 min from Sample 26 (XD-PART3-3U) of 20211015 (recalibrated11).wiff
Max. 3.2e6 cps.
290.1543
291.1569
Intensity, cps
m/z, Da
3s
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C5 H16 N13 O2 | 290.1544 | -0.1434 | -0.4943 | 4.5
2 | C6 H22 N6 O7 | 290.1544 | -0.1488 | -0.5129 | -1
3 | C14 H26 O4 S | 290.1546 | -0.3328 | -1.147 | 2
4 | C20 H20 N O | 290.1539 | 0.3591 | 1.2377 | 11.5
5 | H28 N5 O10 S | 290.1551 | -0.8407 | -2.8976 | -10.5

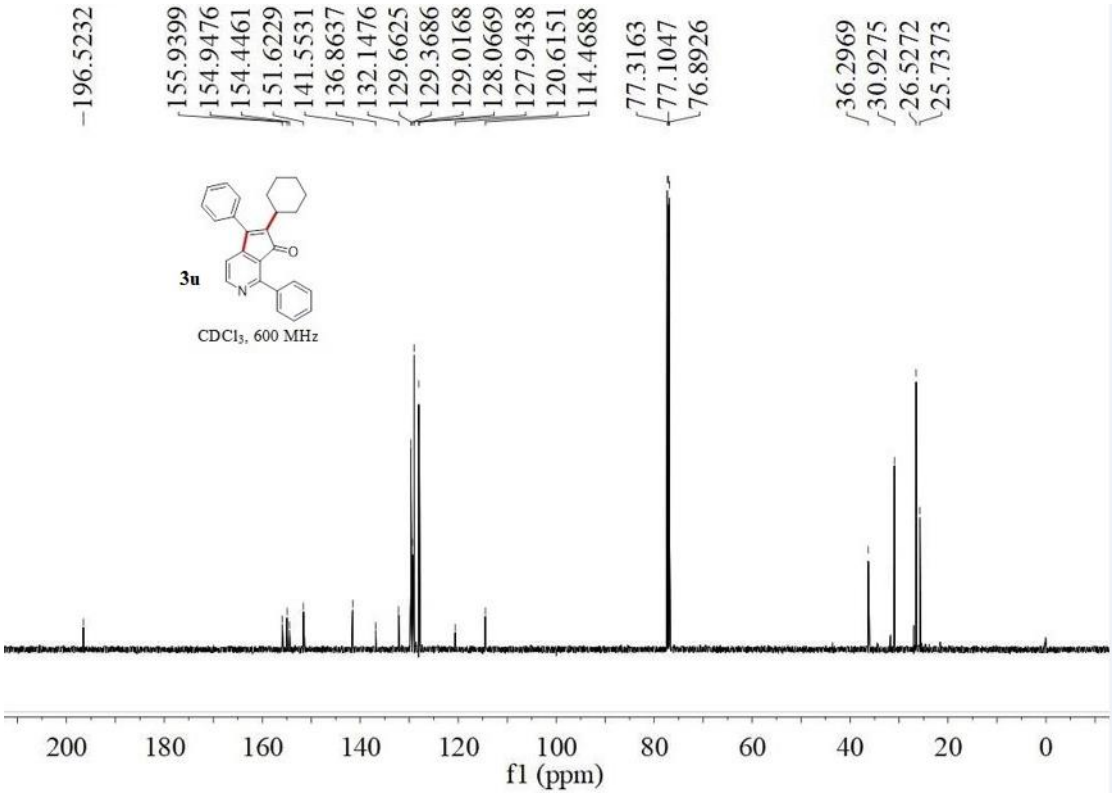

9.2382
8.6403
8.6377
8.6324
8.6298
8.2984
8.2881
8.2851
7.7503
7.7378
7.6919
7.6894
7.6777
7.6761
7.5804
7.5678
7.5579
7.5452
7.5182
7.5059
7.3832
7.3752
7.3699
7.3620
7.2688
1.9934
1.9879
1.9725
1.9670
1.8192
1.7972
1.6842
1.6614
1.2606
1.2388
O
N
N
Ph
3t
CDCl3, 600 MHz
1.00
1.00
1.04
1.01
2.06
3.11
1.04
1.09
1.04
2.05
3.12
2.17
1.19
2.18
10.5
8.0
6.0
4.0
2.0
0.0
f1 (ppm)

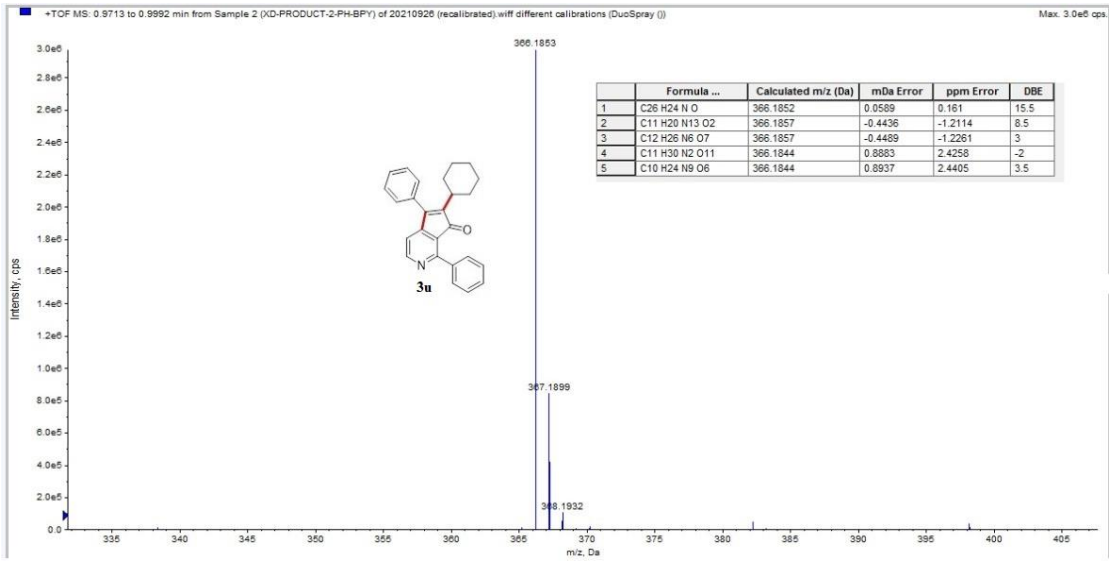

195.7860
167.5478
156.7456
152.6160
150.4078
148.5200
143.5519
134.4259
134.1907
129.5950
129.4402
129.4231
128.3818
124.4492
123.6149
118.3208
77.3242
77.1125
76.9009
36.6764
30.9713
26.5265
25.7800
O
N
N
Ph
3t
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

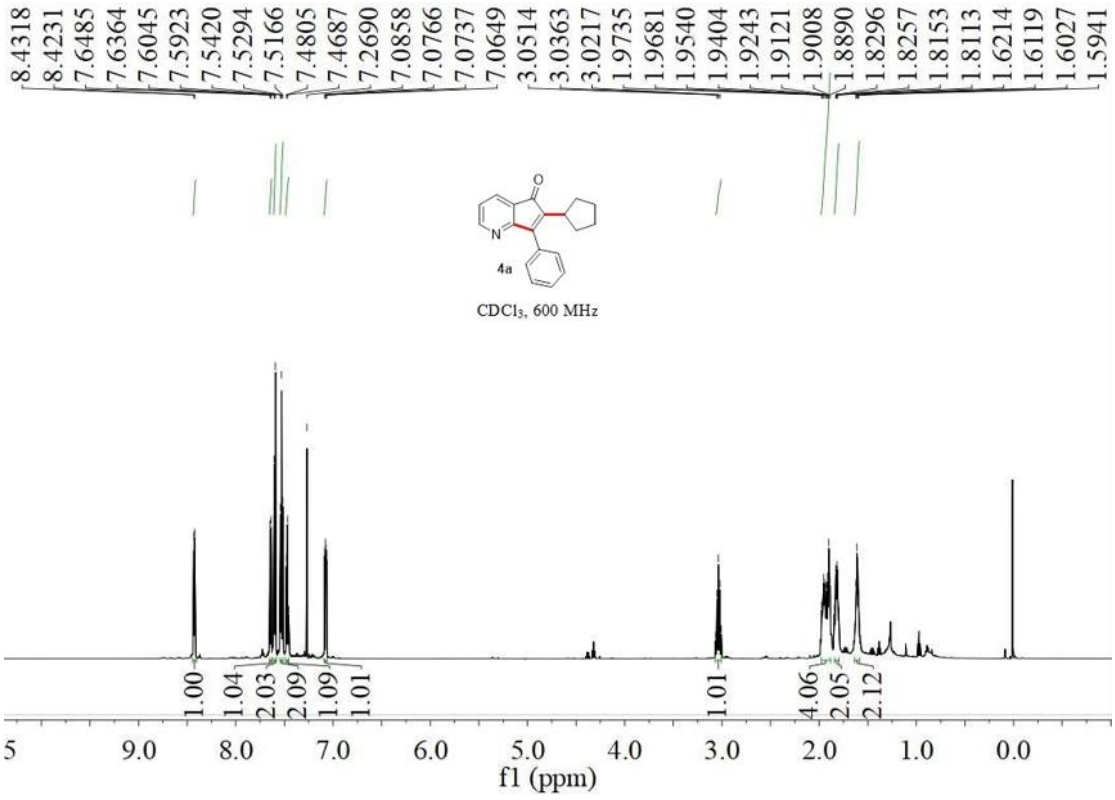


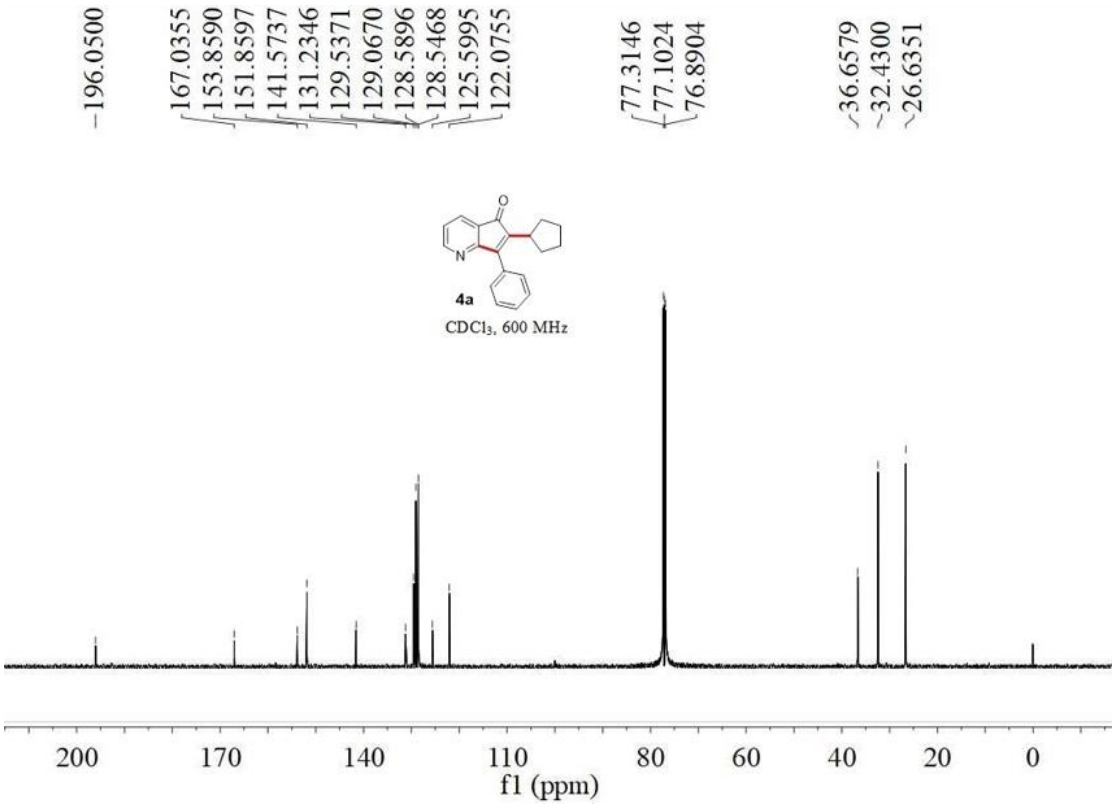
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C25 H23 N2 O	367.1804	0.11	0.2995	15.5
2	C10 H19 N14 O2	367.1809	-0.3925	-1.0691	8.5
3	C11 H25 N7 O7	367.1809	-0.3979	-1.0838	3
4	C9 H23 N10 O6	367.1796	0.9447	2.5729	3.5
5	C8 H17 N17 O	367.1796	0.9501	2.5876	9

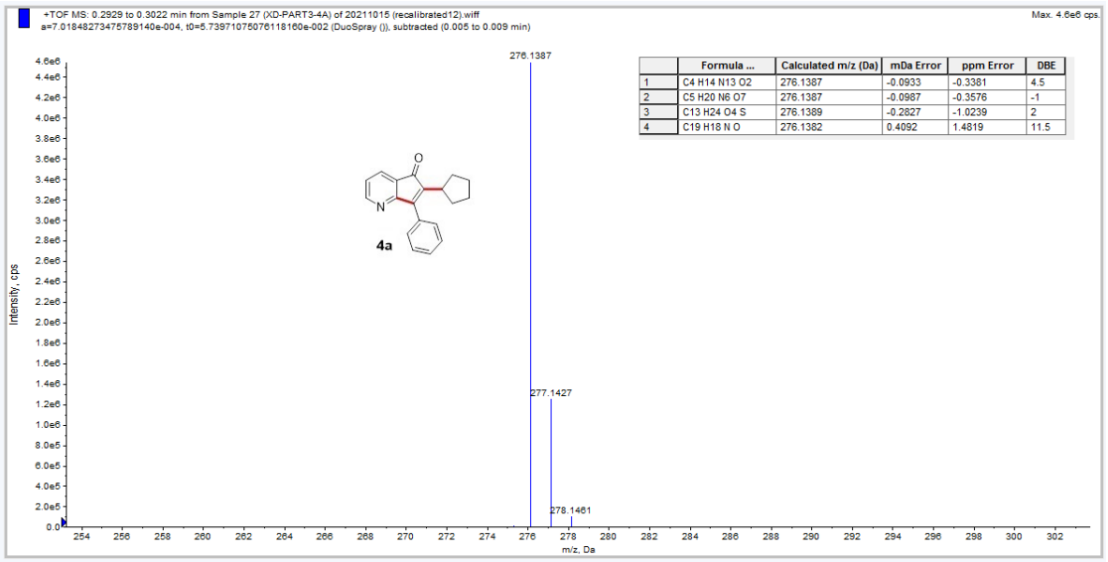


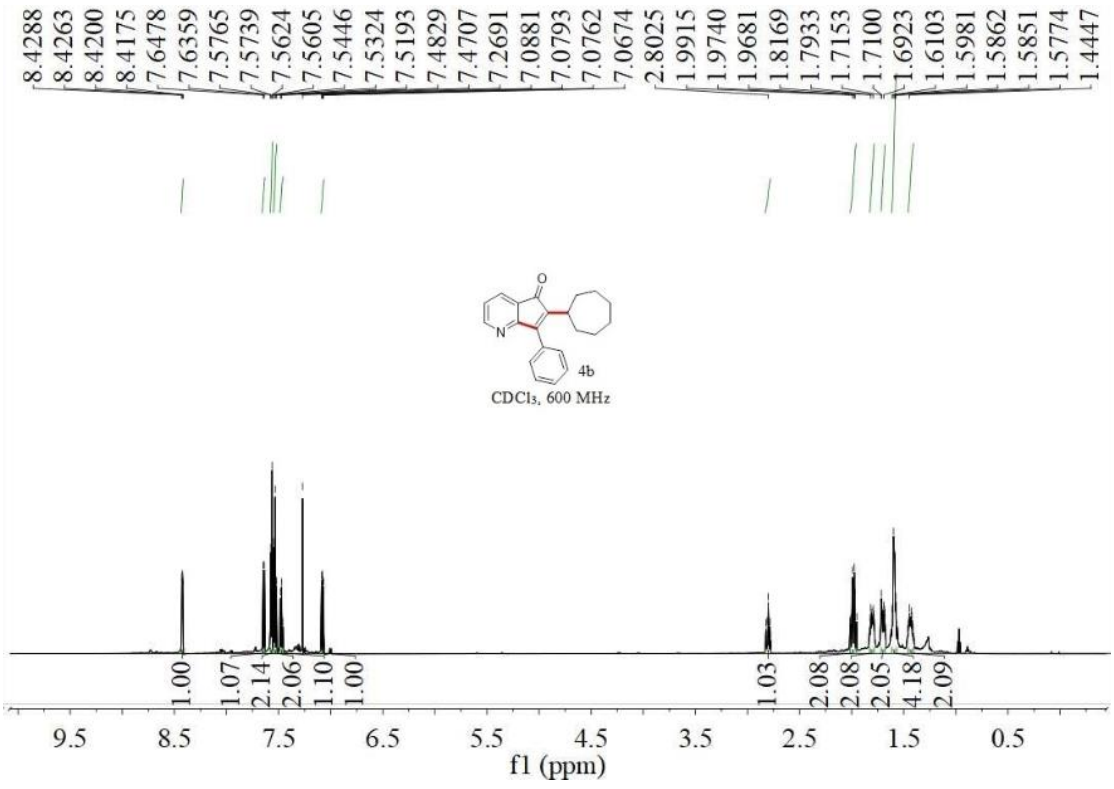

196.5232
155.9399
154.9476
154.4461
151.6229
141.5531
136.8637
132.1476
129.6625
129.3686
129.0168
128.0669
127.9438
120.6151
114.4688
77.3163
77.1047
76.8926
36.2969
30.9275
26.5272
25.7373
3u
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

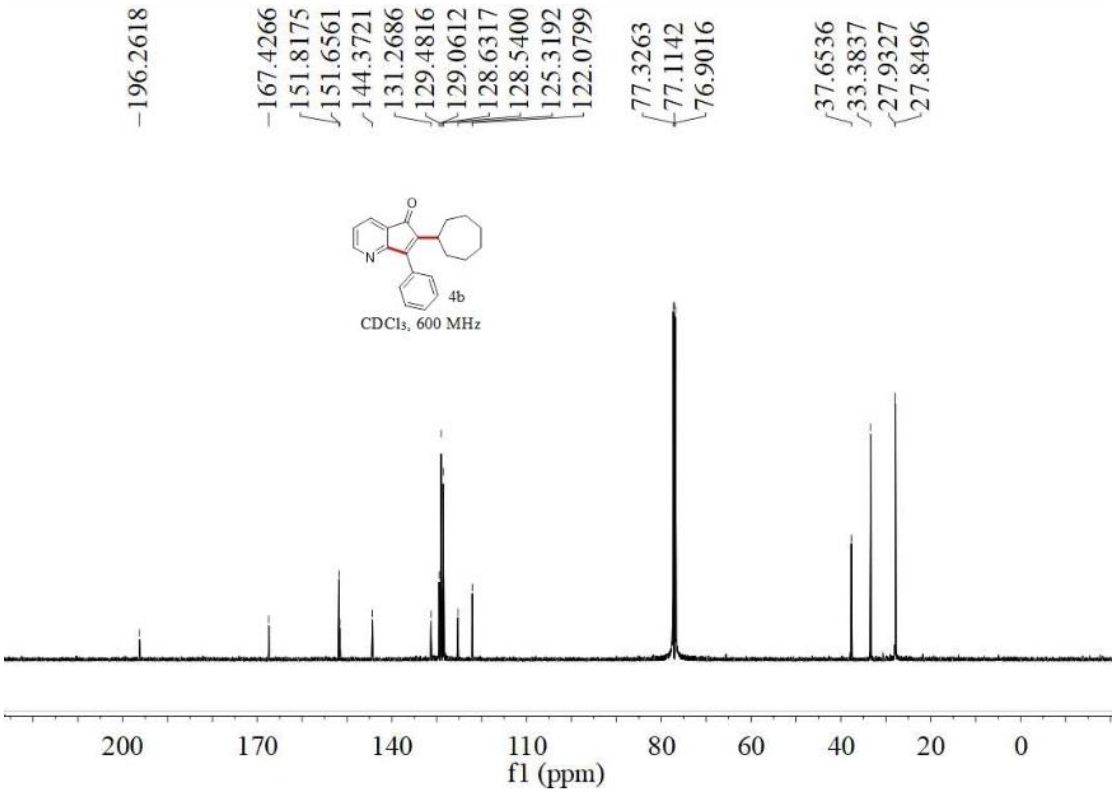

+TOF MS: 0.9713 to 0.9992 min from Sample 2 (XD-PRODUCT-2-PH-BPY) of 20210926 (recalibrated).wiff different calibrations (DuoSpray ())
Max. 3.0e6 cps.
366.1853
367.1899
368.1932
3u
Intensity, cps
m/z, Da
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C26 H24 N O | 366.1852 | 0.0589 | 0.161 | 15.5
2 | C11 H20 N13 O2 | 366.1857 | -0.4436 | -1.2114 | 8.5
3 | C12 H26 N6 O7 | 366.1857 | -0.4489 | -1.2261 | 3
4 | C11 H30 N2 O11 | 366.1844 | 0.8883 | 2.4258 | -2
5 | C10 H24 N9 O6 | 366.1844 | 0.8937 | 2.4405 | 3.5

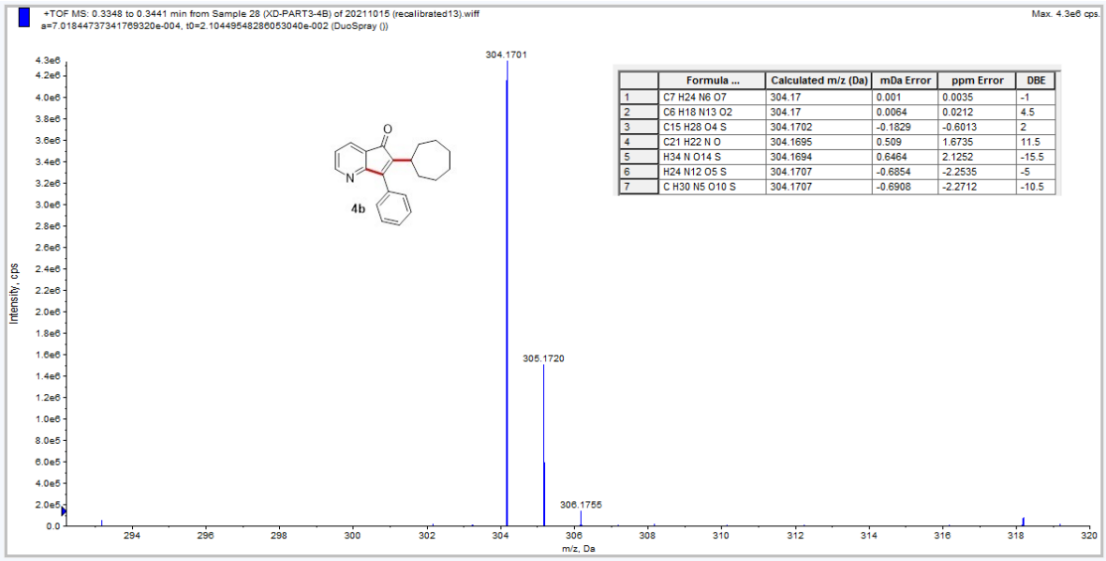

8.4318
8.4231
7.6485
7.6364
7.6045
7.5923
7.5420
7.5294
7.5166
7.4805
7.4687
7.2690
7.0858
7.0766
7.0737
7.0649
3.0514
3.0363
3.0217
1.9735
1.9681
1.9540
1.9404
1.9243
1.9121
1.9008
1.8890
1.8296
1.8257
1.8153
1.8113
1.6214
1.6119
1.6027
1.5941
4a
CDCl3, 600 MHz
1.00
1.04
2.03
2.09
1.09
1.01
1.01
4.06
2.05
2.12
f1 (ppm)

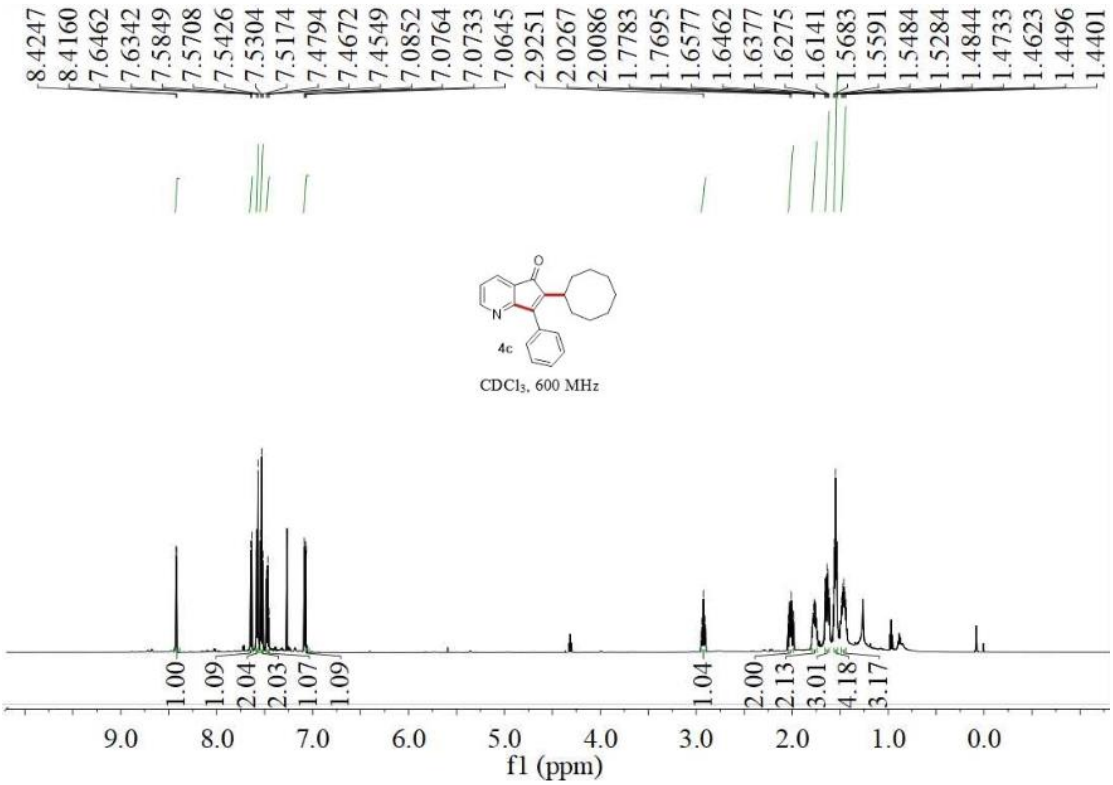

196.0500
167.0355
153.8590
151.8597
141.5737
131.2346
129.5371
129.0670
128.5896
128.5468
125.5995
122.0755
77.3146
77.1024
76.8904
36.6579
32.4300
26.6351
4a
CDCl3, 600 MHz
f1 (ppm)

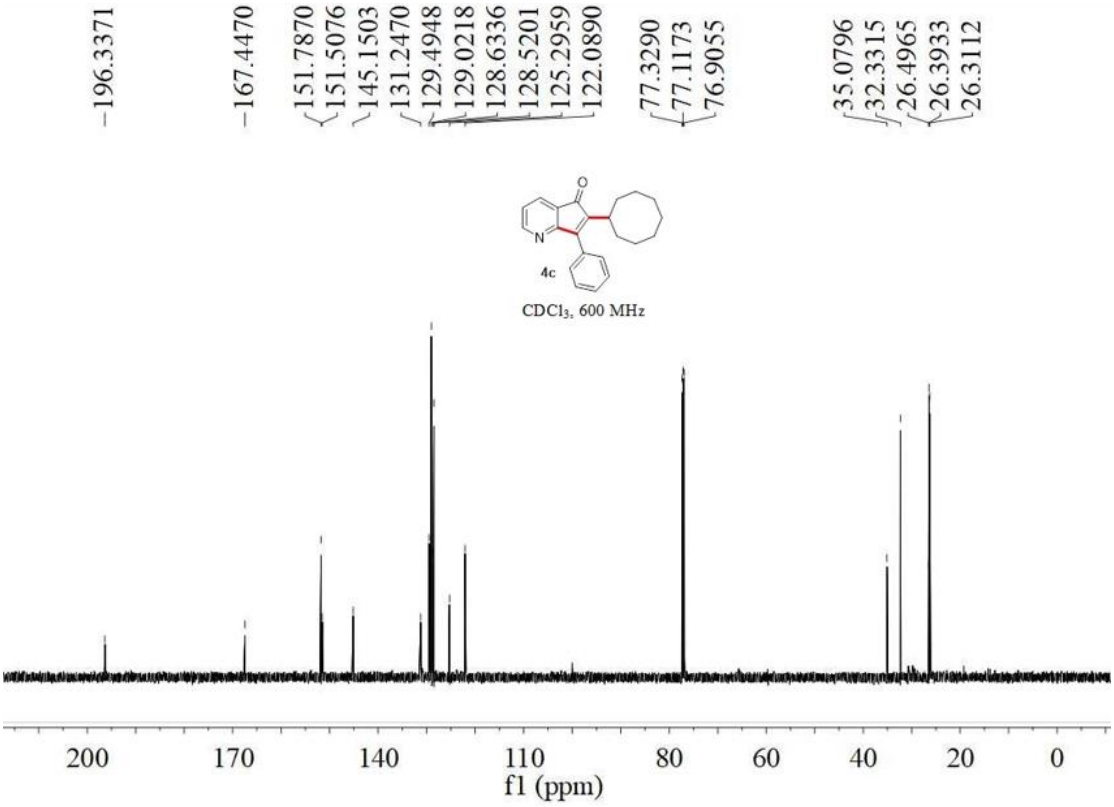

+TOF MS: 0.2929 to 0.3022 min from Sample 27 (XD-PART3-4A) of 20211015 (recalibrated12).wiff
a=7.018482734757891 40e-004, t0=5.739710750761181 60e-002 (DuoSpray ()), subtracted (0.005 to 0.009 min)
Max. 4.6e6 cps
276.1387
277.1427
278.1461
Intensity, cps
m/z, Da
4a
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C4 H14 N13 O2 | 276.1387 | -0.0933 | -0.3381 | 4.5
2 | C5 H20 N6 O7 | 276.1387 | -0.0987 | -0.3576 | -1
3 | C13 H24 O4 S | 276.1389 | -0.2827 | -1.0239 | 2
4 | C19 H18 N O | 276.1382 | 0.4092 | 1.4819 | 11.5

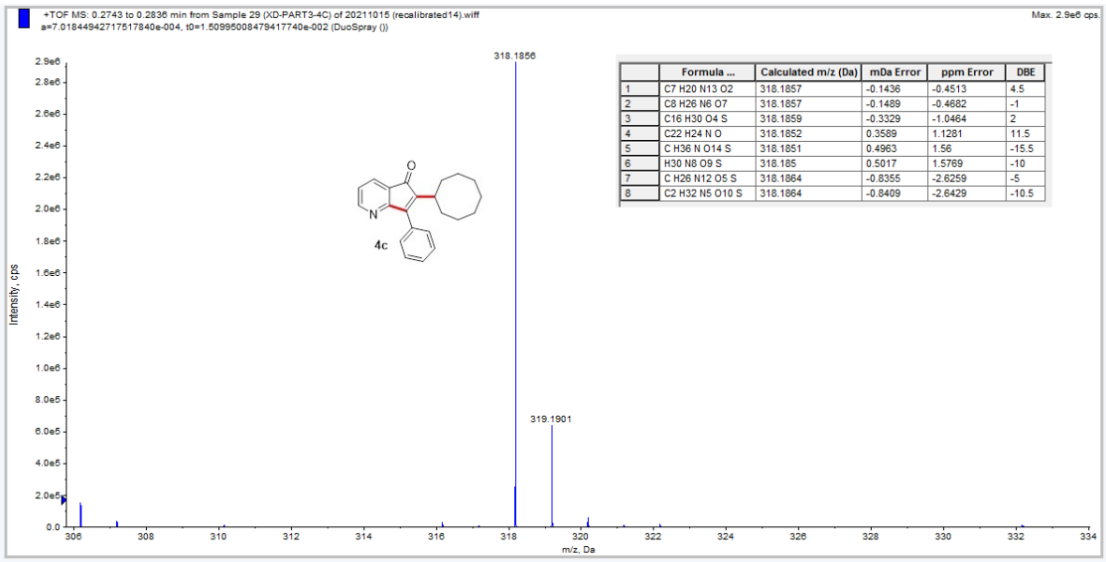

8.4288
8.4263
8.4200
8.4175
7.6478
7.6359
7.5765
7.5739
7.5624
7.5605
7.5446
7.5324
7.5193
7.4829
7.4707
7.2691
7.0881
7.0793
7.0762
7.0674
2.8025
1.9915
1.9740
1.9681
1.8169
1.7933
1.7153
1.7100
1.6923
1.6103
1.5981
1.5862
1.5851
1.5774
1.4447
4b
CDCl3, 600 MHz
1.00
1.07
2.14
2.06
1.10
1.00
1.03
2.08
2.08
2.05
4.18
2.09
f1 (ppm)


196.2618
167.4266
151.8175
151.6561
144.3721
131.2686
129.4816
129.0612
128.6317
128.5400
125.3192
122.0799
77.3263
77.1142
76.9016
37.6536
33.3837
27.9327
27.8496
4b
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

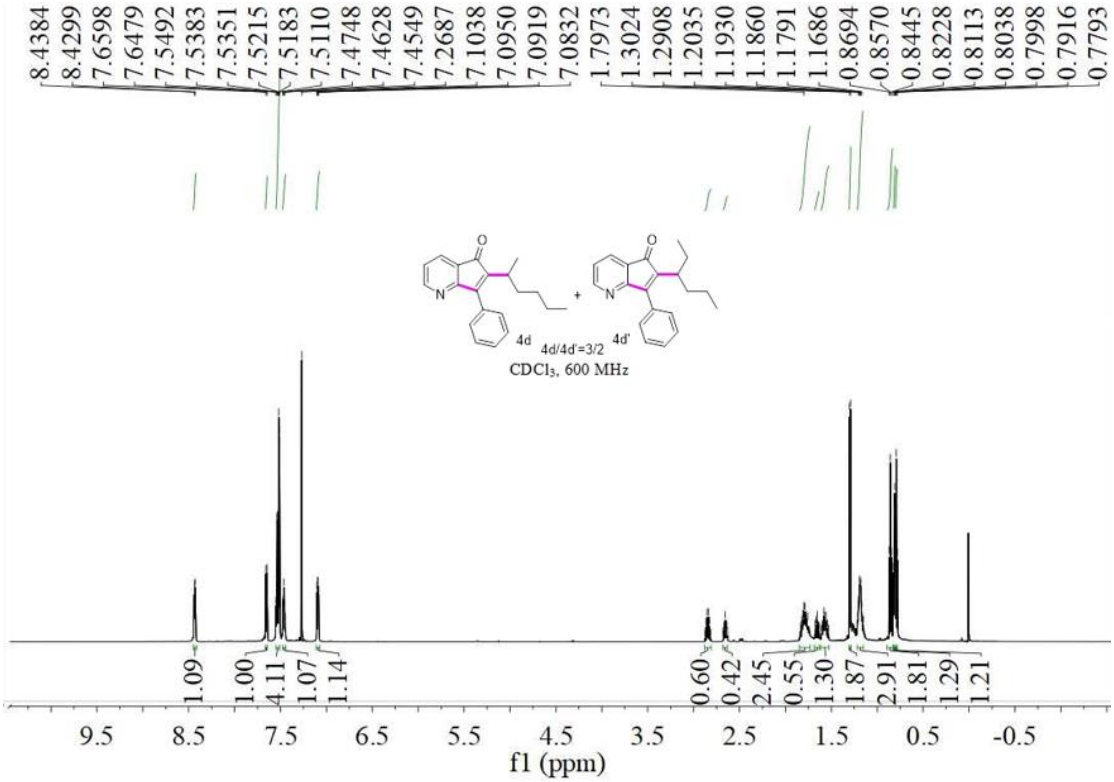

+TOF MS: 0.3348 to 0.3441 min from Sample 28 (XD-PART3-4B) of 20211015 (recalibrated13).wiff
a=7.0184473734176932e-004, t0=2.1044954828605304e-002 (DuoSpray ())
Max. 4.3e6 cps
304.1701
305.1720
306.1755
4b
Intensity, cps
m/z, Da
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C7 H24 N6 O7 | 304.17 | 0.001 | 0.0035 | -1
2 | C6 H18 N13 O2 | 304.17 | 0.0064 | 0.0212 | 4.5
3 | C15 H28 O4 S | 304.1702 | -0.1829 | -0.6013 | 2
4 | C21 H22 N O | 304.1695 | 0.509 | 1.6735 | 11.5
5 | H34 N O14 S | 304.1694 | 0.6464 | 2.1252 | -15.5
6 | H24 N12 O5 S | 304.1707 | -0.6854 | -2.2535 | -5
7 | C H30 N5 O10 S | 304.1707 | -0.6908 | -2.2712 | -10.5

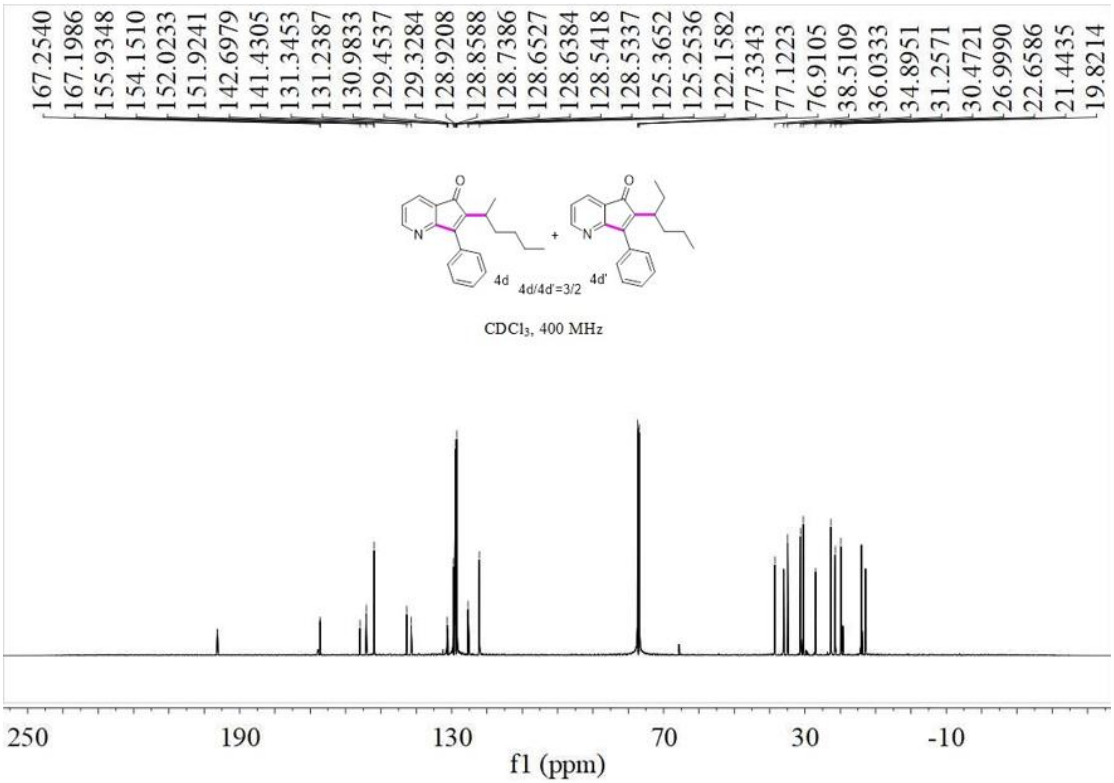

8.4247
8.4160
7.6462
7.6342
7.5849
7.5708
7.5426
7.5304
7.5174
7.4794
7.4672
7.4549
7.0852
7.0764
7.0733
7.0645
2.9251
2.0267
2.0086
1.7783
1.7695
1.6577
1.6462
1.6377
1.6275
1.6141
1.5683
1.5591
1.5484
1.5284
1.4844
1.4733
1.4623
1.4496
1.4401
4c
CDCl3, 600 MHz
1.00
1.09
2.04
2.03
1.07
1.09
1.04
2.00
2.13
3.01
4.18
3.17
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

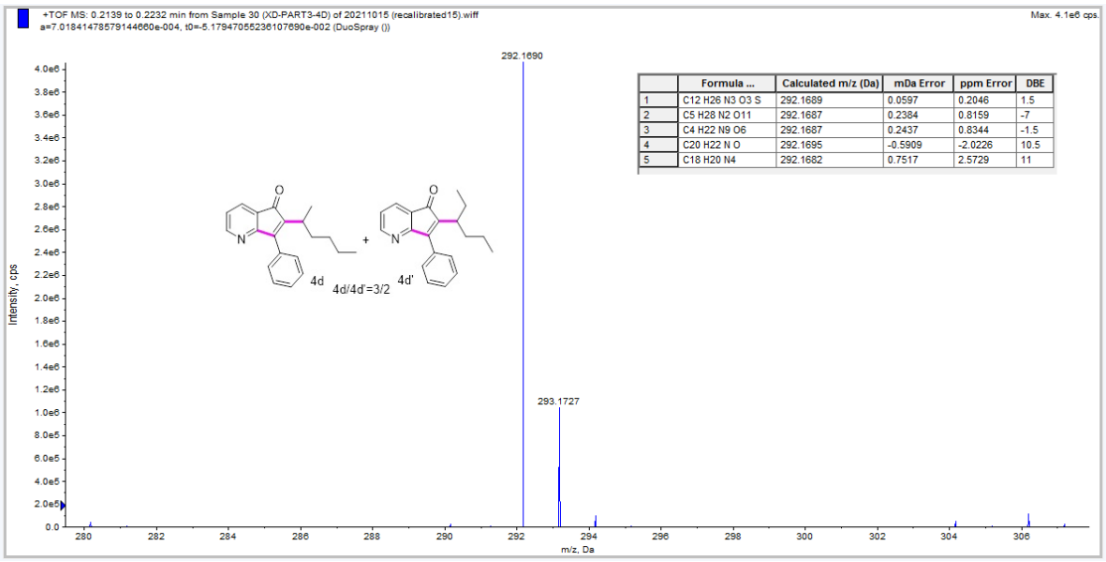

196.3371
167.4470
151.7870
151.5076
145.1503
131.2470
129.4948
129.0218
128.6336
128.5201
125.2959
122.0890
77.3290
77.1173
76.9055
35.0796
32.3315
26.4965
26.3933
26.3112
4c
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

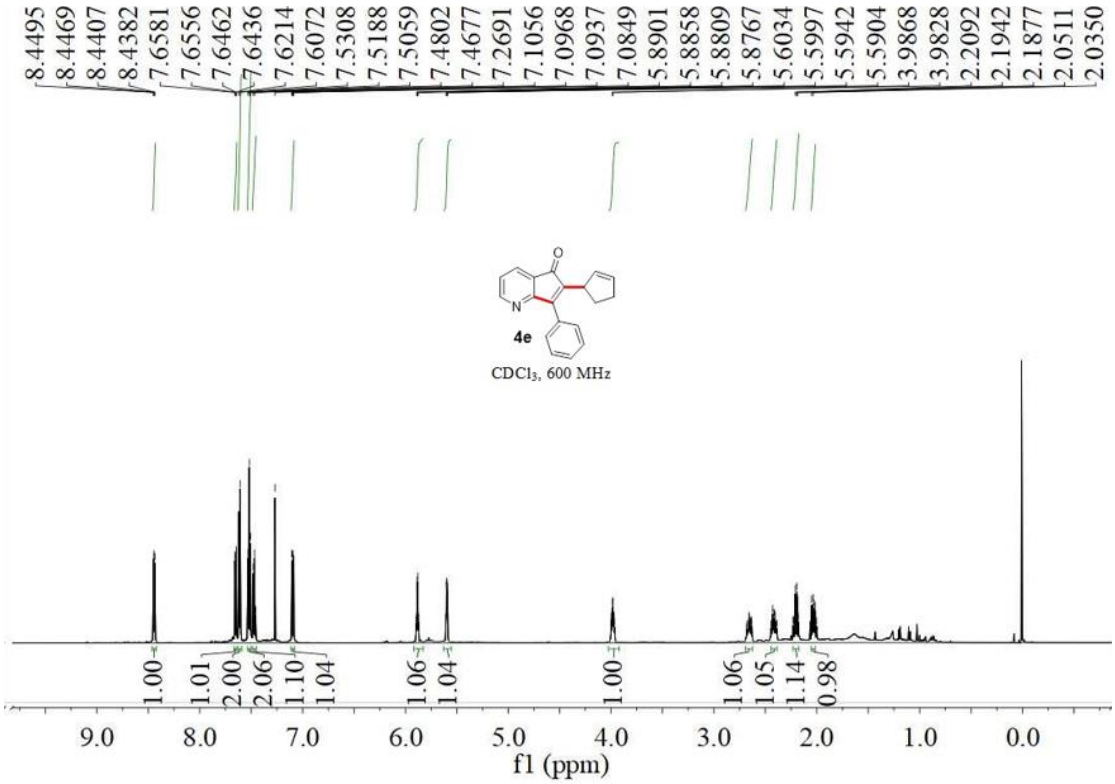


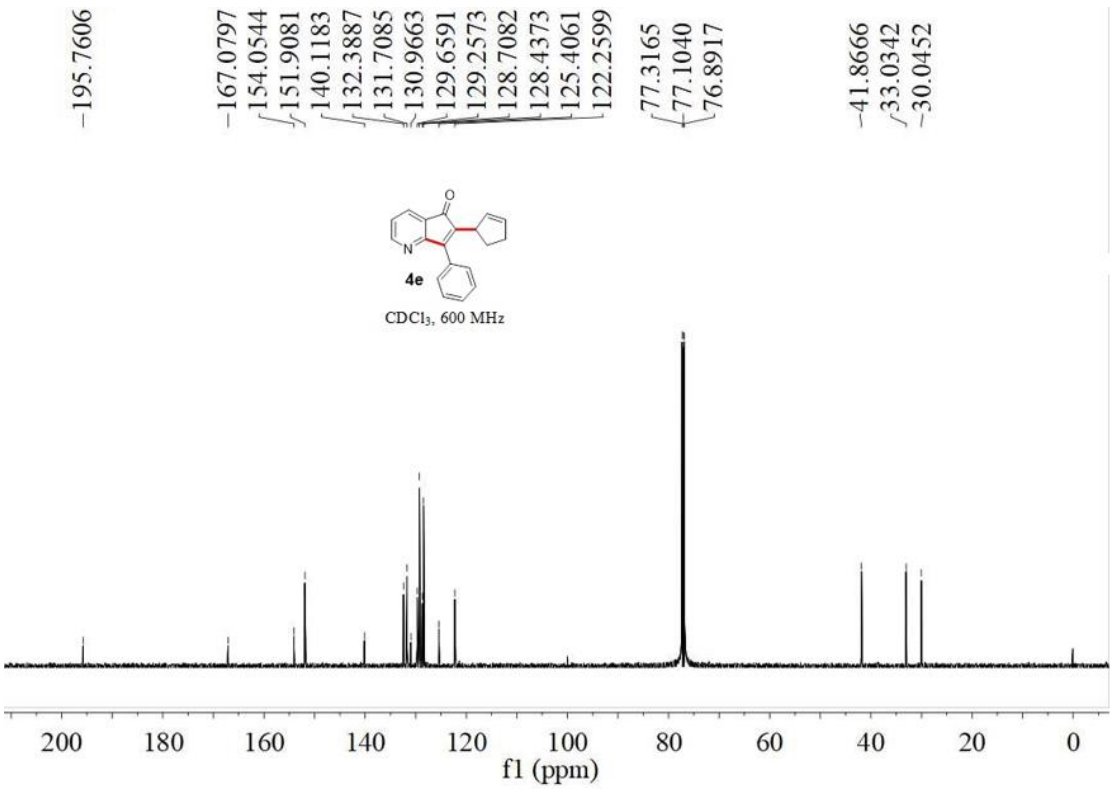
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C7 H20 N13 O2	318.1857	-0.1436	-0.4513	4.5
2	C8 H26 N6 O7	318.1857	-0.1489	-0.4682	-1
3	C16 H30 O4 S	318.1859	-0.3329	-1.0464	2
4	C22 H24 N O	318.1852	0.3589	1.1281	11.5
5	C H36 N O14 S	318.1851	0.4963	1.56	-15.5
6	H30 N8 O9 S	318.185	0.5017	1.5769	-10
7	C H26 N12 O5 S	318.1864	-0.8355	-2.6259	-5
8	C2 H32 N5 O10 S	318.1864	-0.8409	-2.6429	-10.5

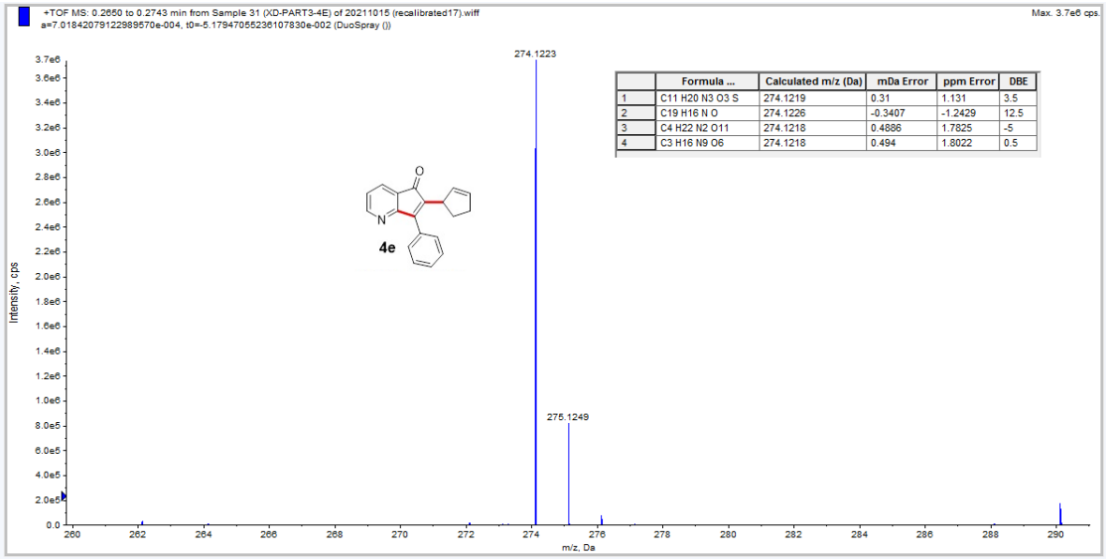


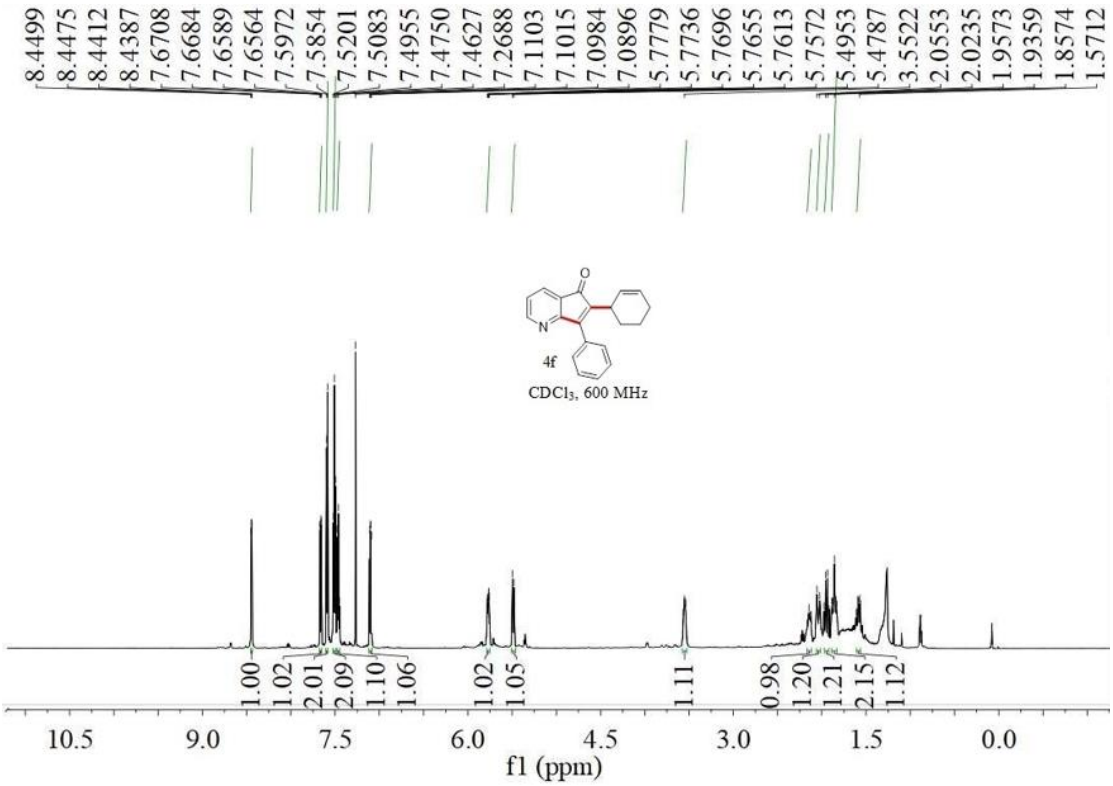

167.2540
167.1986
155.9348
154.1510
152.0233
151.9241
142.6979
141.4305
131.3453
131.2387
130.9833
129.4537
129.3284
128.9208
128.8588
128.7386
128.6527
128.6384
128.5418
128.5337
125.3652
125.2536
122.1582
77.3343
77.1223
76.9105
38.5109
36.0333
34.8951
31.2571
30.4721
26.9990
22.6586
21.4435
19.8214
4d
4d/4d'=3/2
4d'
CDCl3, 400 MHz
250
190
130
70
30
-10
f1 (ppm)

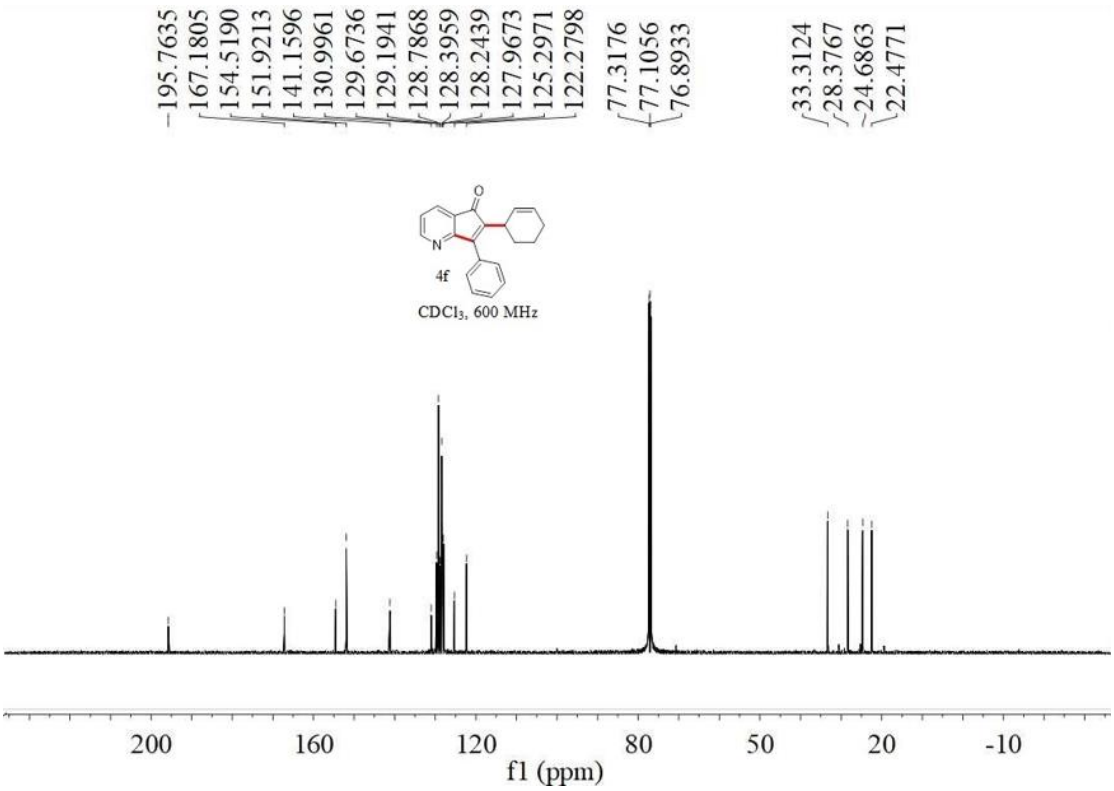

+TOF MS: 0.2139 to 0.2232 min from Sample 30 (XD-PART3-4D) of 20211015 (recalibrated15).wiff
a=7.0184147857914466e-004, t0=-5.1794705523610769e-002 (DuoSpray ())
Max. 4.1e6 cps.
292.1690
293.1727
Intensity, cps
m/z, Da
4d
4d/4d'=3/2
4d'
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C12 H26 N3 O3 S | 292.1689 | 0.0597 | 0.2046 | 1.5
2 | C5 H28 N2 O11 | 292.1687 | 0.2384 | 0.8159 | -7
3 | C4 H22 N9 O6 | 292.1687 | 0.2437 | 0.8344 | -1.5
4 | C20 H22 N O | 292.1695 | -0.5909 | -2.0226 | 10.5
5 | C18 H20 N4 | 292.1682 | 0.7517 | 2.5729 | 11

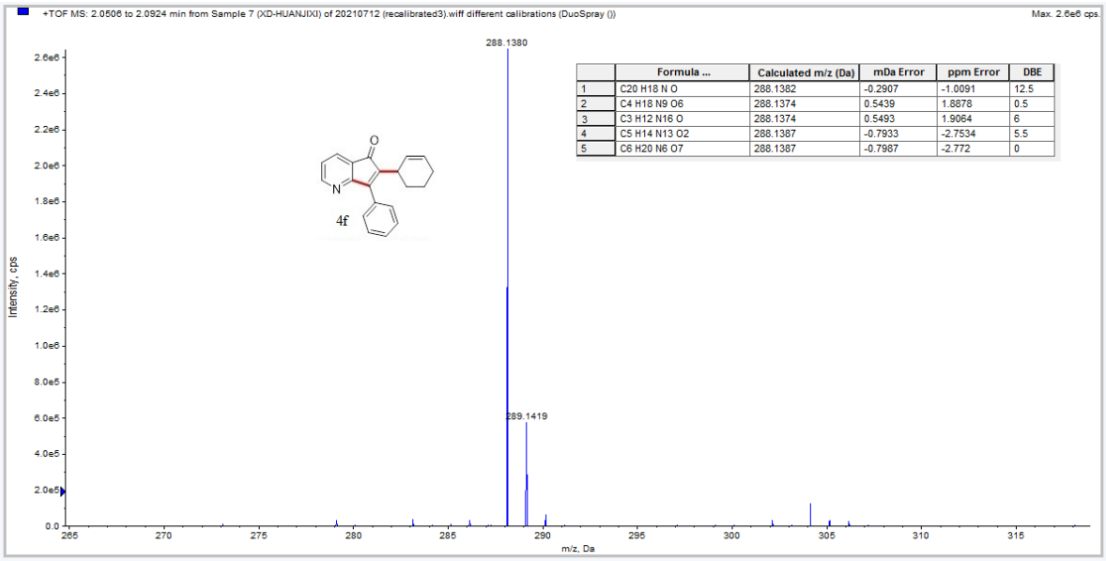

8.4495
8.4469
8.4407
8.4382
7.6581
7.6556
7.6462
7.6436
7.6214
7.6072
7.5308
7.5188
7.5059
7.4802
7.4677
7.2691
7.1056
7.0968
7.0937
7.0849
5.8901
5.8858
5.8809
5.8767
5.6034
5.5997
5.5942
5.5904
3.9868
3.9828
2.2092
2.1942
2.1877
2.0511
2.0350
4e
CDCl3, 600 MHz
1.00
1.01
2.00
2.06
1.10
1.04
1.06
1.04
1.00
1.06
1.05
1.14
0.98
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

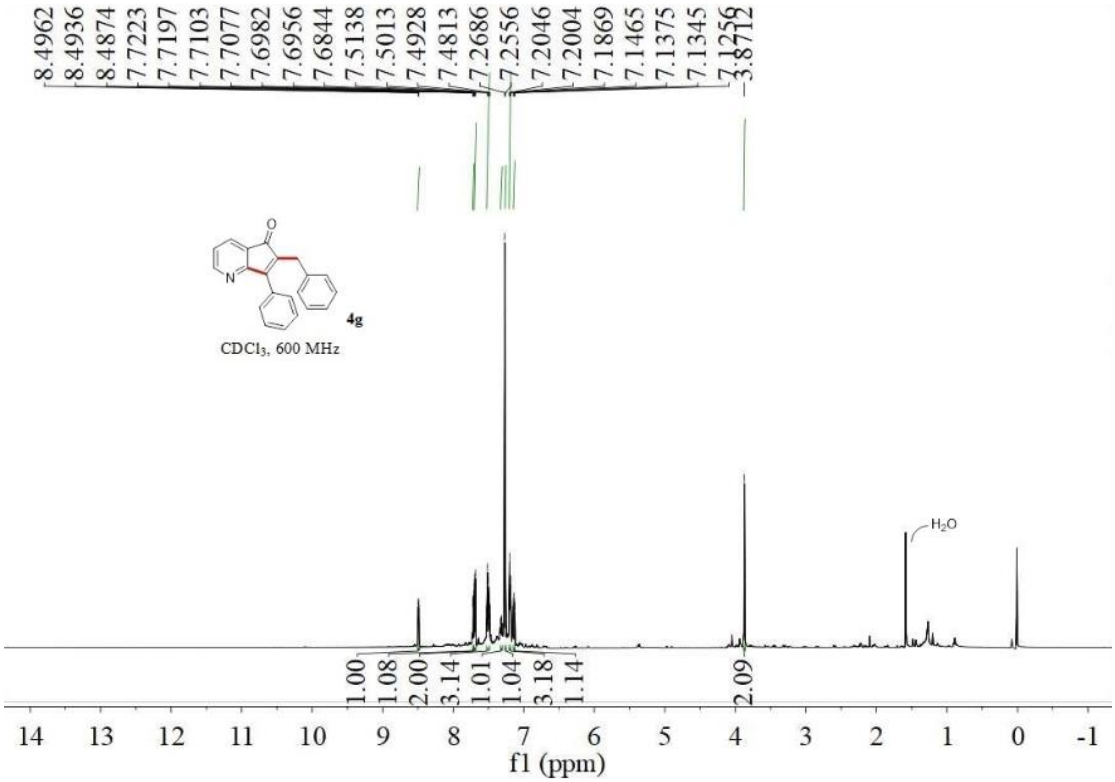

195.7606
167.0797
154.0544
151.9081
140.1183
132.3887
131.7085
130.9663
129.6591
129.2573
128.7082
128.4373
125.4061
122.2599
77.3165
77.1040
76.8917
41.8666
33.0342
30.0452
4e
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

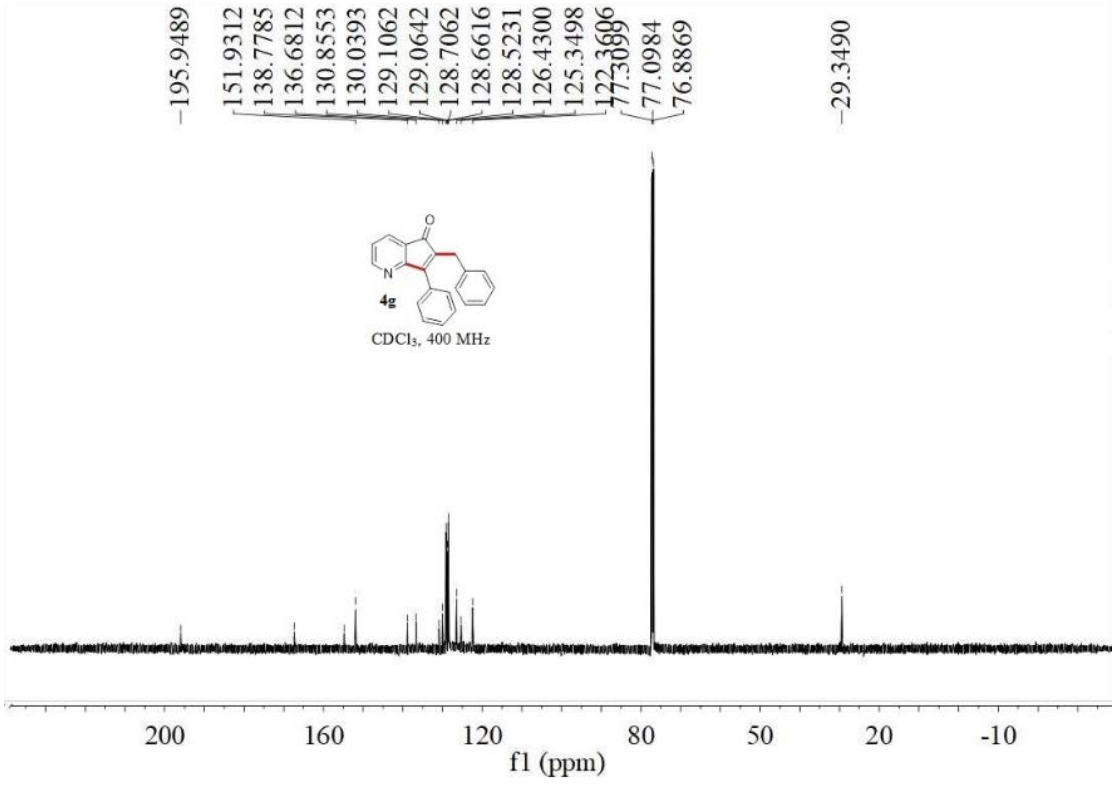

+TOF MS: 0.2650 to 0.2743 min from Sample 31 (XD-PART3-4E) of 20211015 (recalibrated17).wiff
a=7.018420791229895 70e-004, t0=-5.179470552361078 30e-002 (DuoSpray ())
Max. 3.7e6 cps.
274.1223
275.1249
Intensity, cps
m/z, Da
4e
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C11 H20 N3 O3 S | 274.1219 | 0.31 | 1.131 | 3.5
2 | C19 H16 N O | 274.1226 | -0.3407 | -1.2429 | 12.5
3 | C4 H22 N2 O11 | 274.1218 | 0.4886 | 1.7825 | -5
4 | C3 H16 N9 O6 | 274.1218 | 0.494 | 1.8022 | 0.5

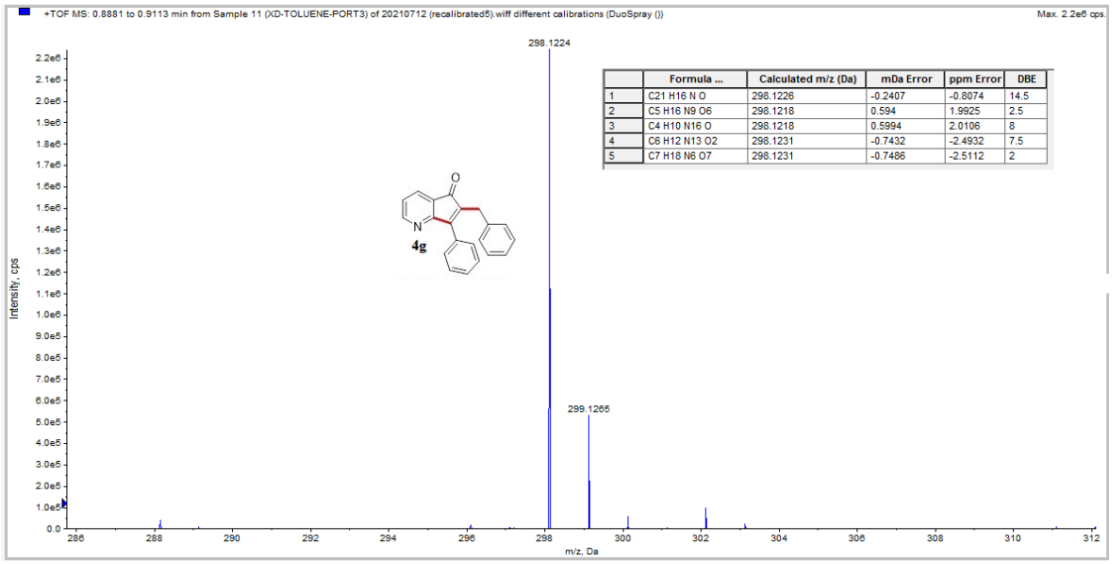

8.4499
8.4475
8.4412
8.4387
7.6708
7.6684
7.6589
7.6564
7.5972
7.5854
7.5201
7.5083
7.4955
7.4750
7.4627
7.2688
7.1103
7.1015
7.0984
7.0896
5.7779
5.7736
5.7696
5.7655
5.7613
5.7572
5.4953
5.4787
3.5522
2.0553
2.0235
1.9573
1.9359
1.8574
1.5712
4f
CDCl3, 600 MHz
1.00
1.02
2.01
2.09
1.10
1.06
1.02
1.05
1.11
0.98
1.20
1.21
2.15
1.12
10.5
9.0
7.5
6.0
4.5
3.0
1.5
0.0
f1 (ppm)


195.7635
167.1805
154.5190
151.9213
141.1596
130.9961
129.6736
129.1941
128.7868
128.3959
128.2439
127.9673
125.2971
122.2798
77.3176
77.1056
76.8933
33.3124
28.3767
24.6863
22.4771
4f
CDCl3, 600 MHz
200
160
120
80
50
20
-10
f1 (ppm)

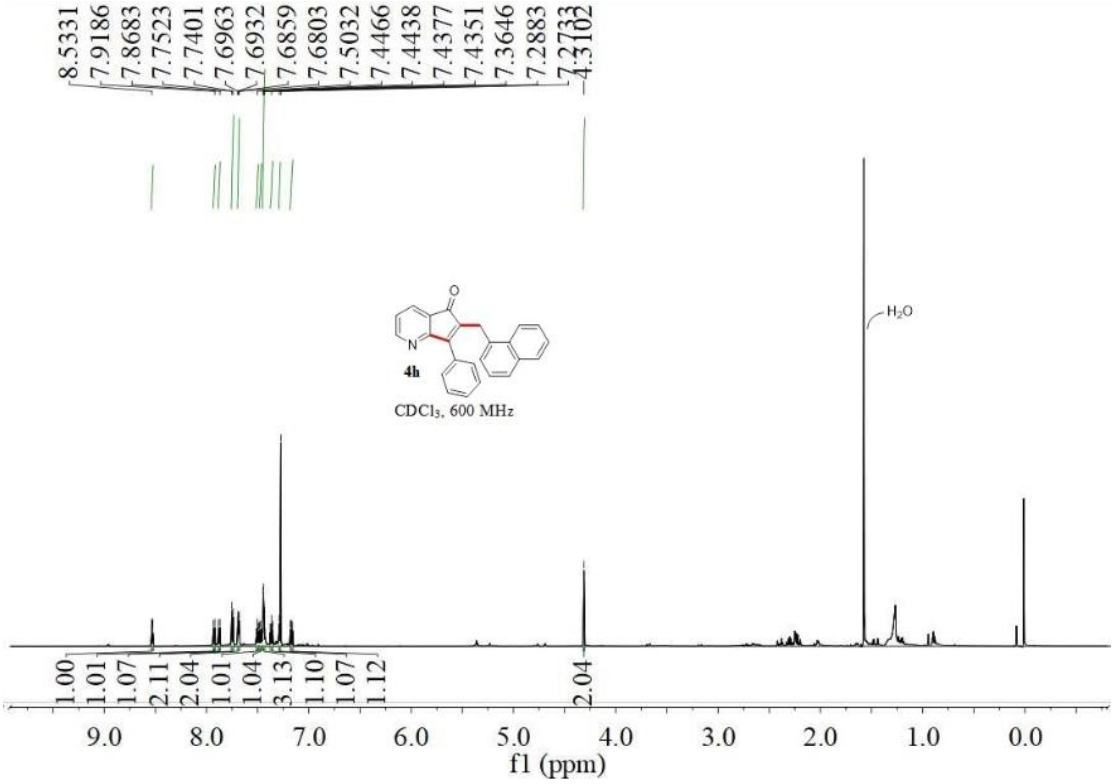

+TOF MS: 2.0506 to 2.0924 min from Sample 7 (XD-HUANJIXI) of 20210712 (recalibrated3).wiff different calibrations (DuoSpray ())
Max. 2.6e6 cps.
288.1380
289.1419
4f
Intensity, cps
m/z, Da
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C20 H18 N O | 288.1382 | -0.2907 | -1.0091 | 12.5
2 | C4 H18 N9 O6 | 288.1374 | 0.5439 | 1.8878 | 0.5
3 | C3 H12 N16 O | 288.1374 | 0.5493 | 1.9064 | 6
4 | C5 H14 N13 O2 | 288.1387 | -0.7933 | -2.7534 | 5.5
5 | C6 H20 N6 O7 | 288.1387 | -0.7987 | -2.772 | 0

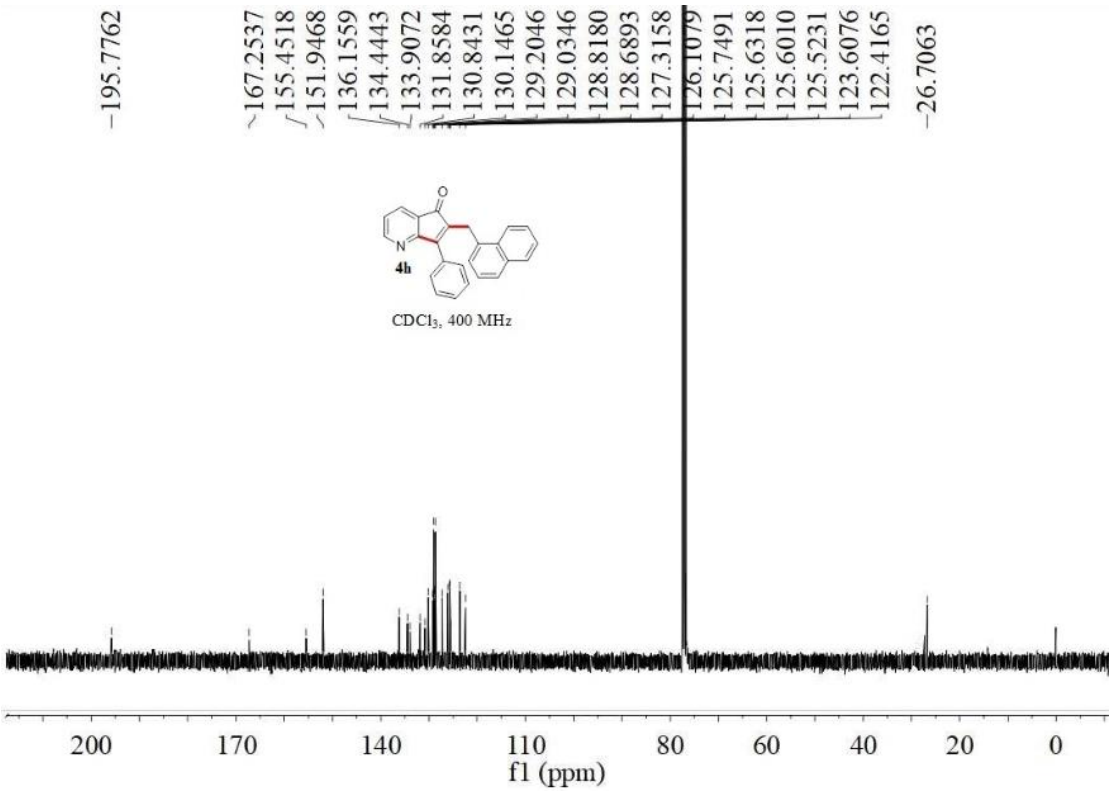

8.4962
8.4936
8.4874
7.7223
7.7197
7.7103
7.7077
7.6982
7.6956
7.6844
7.5138
7.5013
7.4928
7.4813
7.2686
7.2556
7.2046
7.2004
7.1869
7.1465
7.1375
7.1345
7.1256
3.8712
4g
CDCl3, 600 MHz
H2O
1.00
1.08
2.00
3.14
1.01
1.04
3.18
1.14
2.09
14 13 12 11 10 9 8 7 6 5 4 3 2 1 0 -1
f1 (ppm)

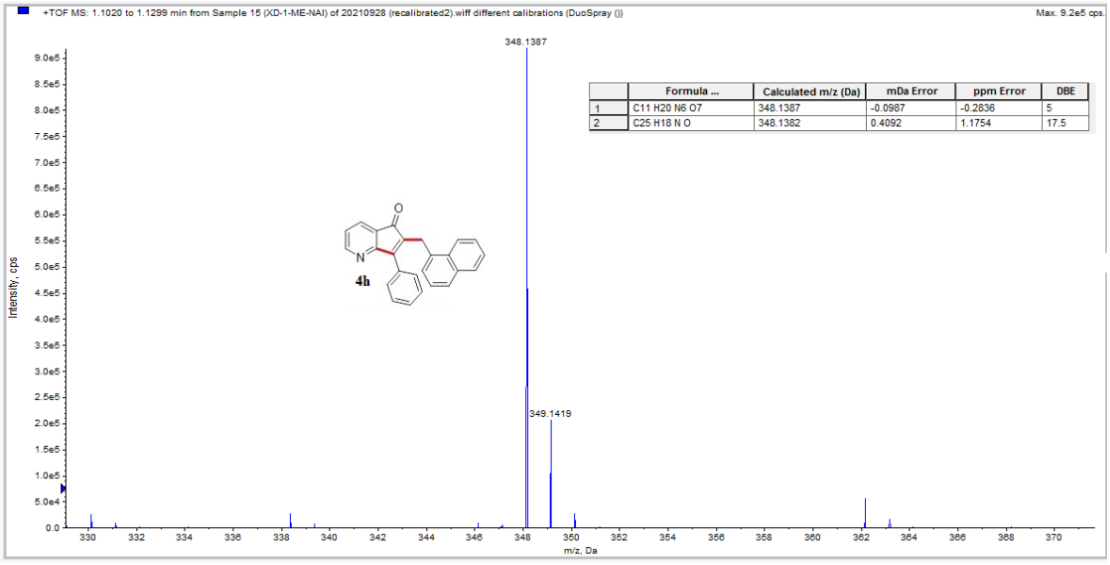

195.9489
151.9312
138.7785
136.6812
130.8553
130.0393
129.1062
129.0642
128.7062
128.6616
128.5231
126.4300
125.3498
122.3606
77.3089
77.0984
76.8869
29.3490
4g
CDCl3, 400 MHz
200 160 120 80 50 20 -10
f1 (ppm)

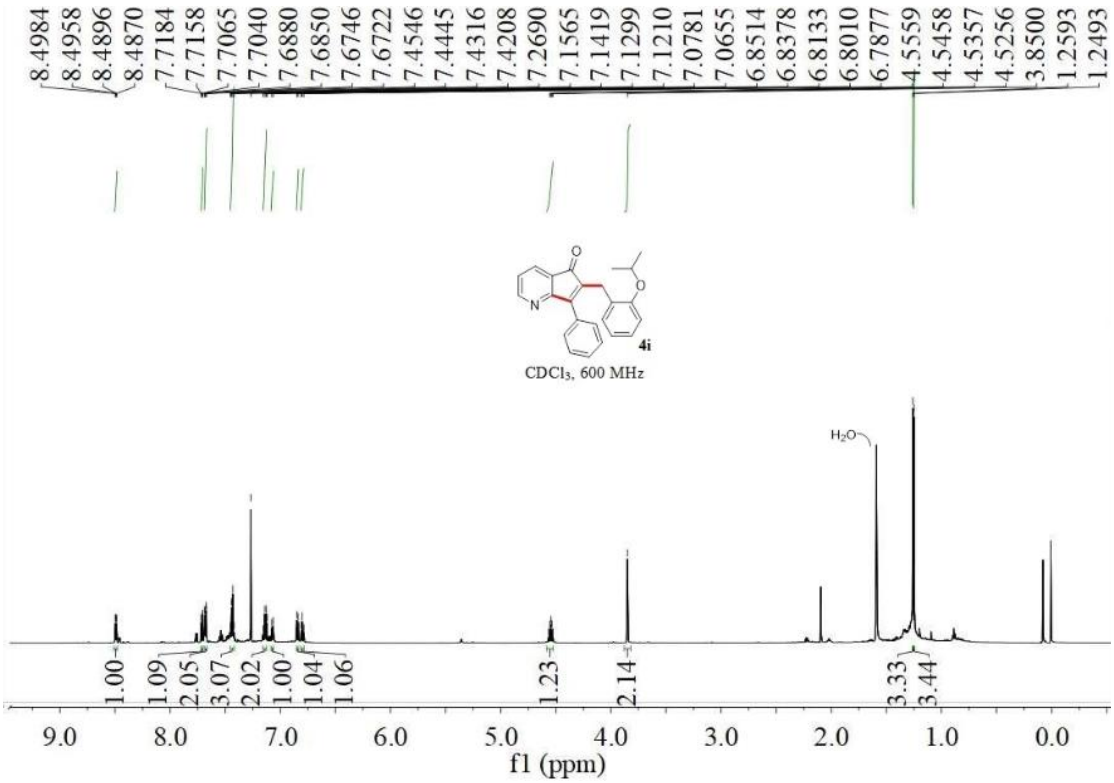


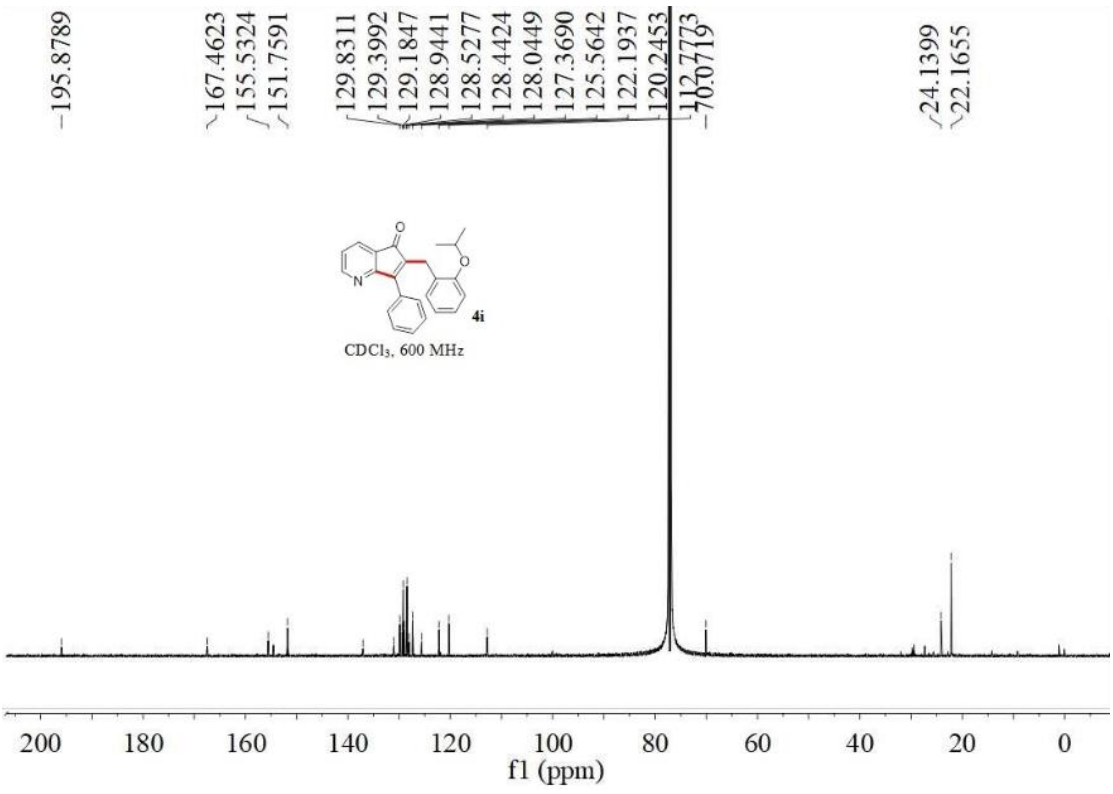
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C21 H16 N O	298.1226	-0.2407	-0.8074	14.5
2	C5 H16 N9 O6	298.1218	0.594	1.9925	2.5
3	C4 H10 N16 O	298.1218	0.5994	2.0106	8
4	C6 H12 N13 O2	298.1231	-0.7432	-2.4932	7.5
5	C7 H18 N6 O7	298.1231	-0.7486	-2.5112	2

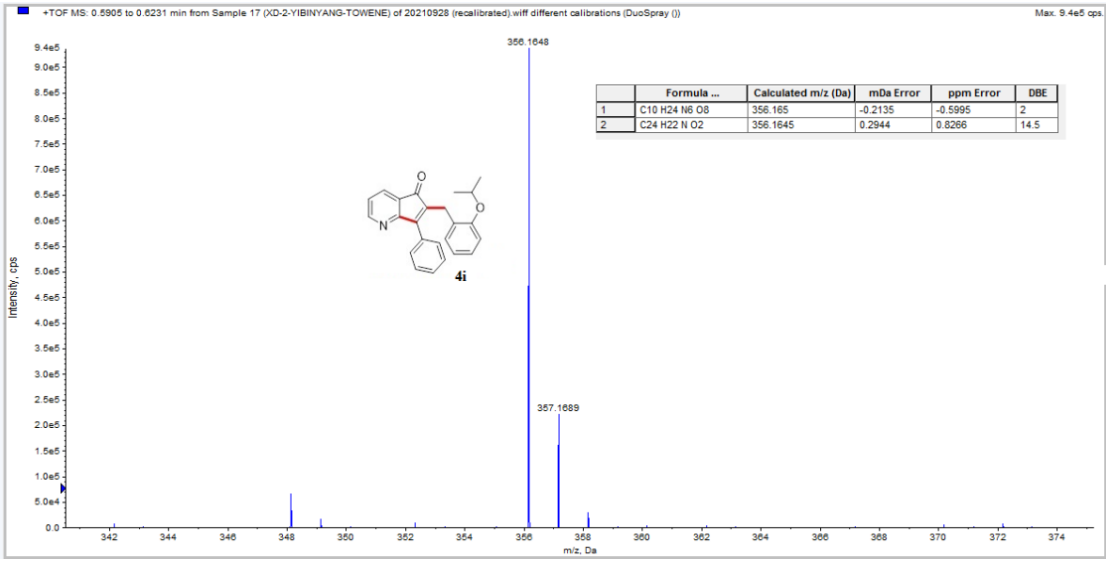


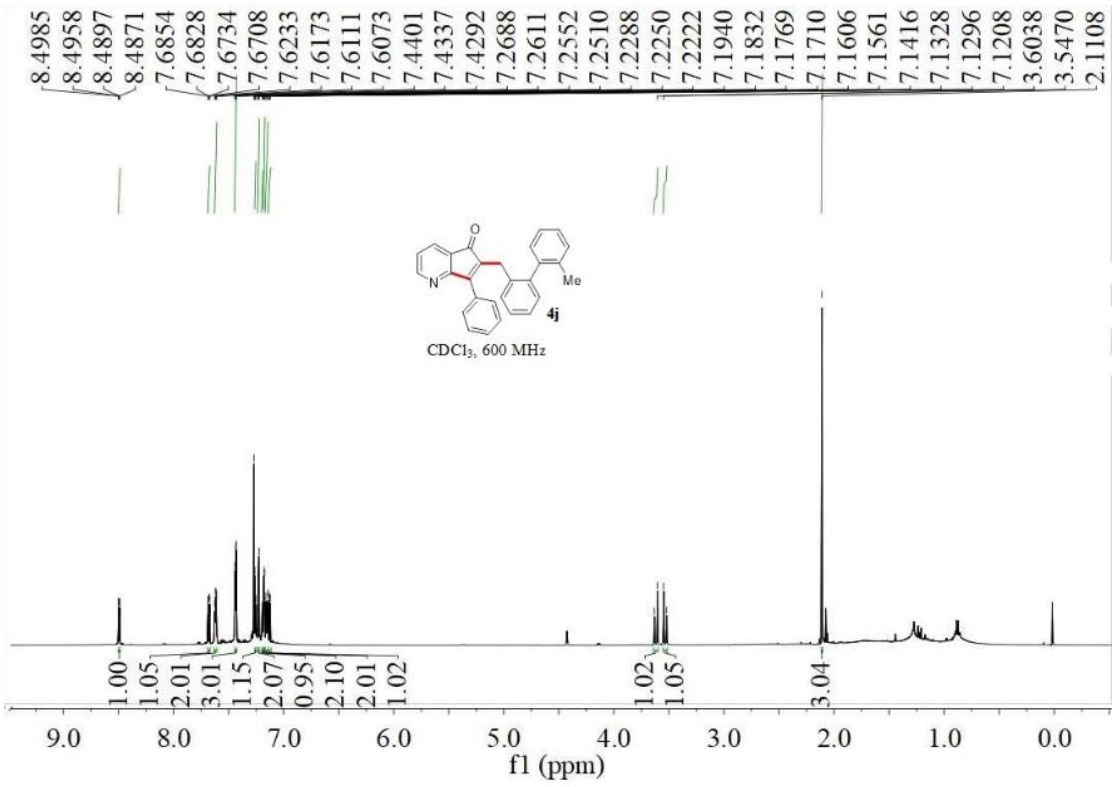

195.7762
167.2537
155.4518
151.9468
136.1559
134.4443
133.9072
131.8584
130.8431
130.1465
129.2046
129.0346
128.8180
128.6893
127.3158
126.1079
125.7491
125.6318
125.6010
125.5231
123.6076
122.4165
26.7063
4h
CDCl3, 400 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

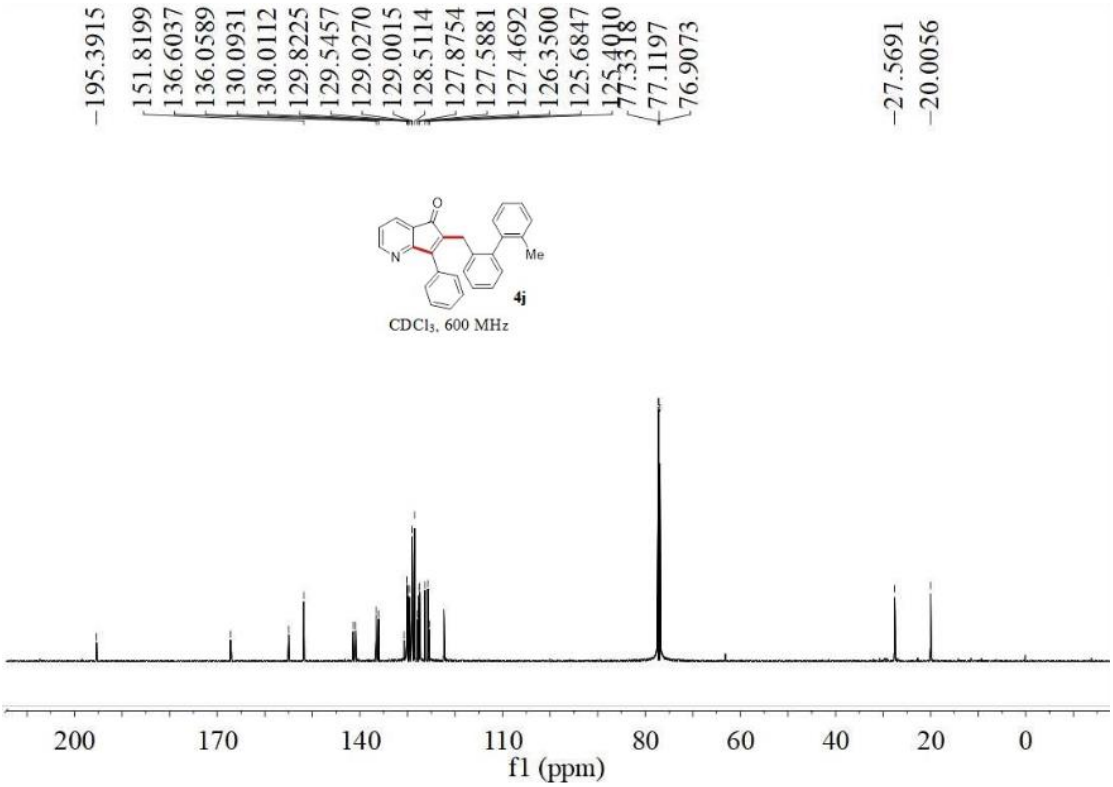

+TOF MS: 1.1020 to 1.1299 min from Sample 15 (XD-1-ME-NAI) of 20210928 (recalibrated2).wiff different calibrations (DuoSpray ())
Max. 9.2e5 cps.
348.1387
349.1419
Intensity, cps
m/z, Da
4h
Formula ...
Calculated m/z (Da)
mDa Error
ppm Error
DBE
1
C11 H20 N6 O7
348.1387
-0.0987
-0.2836
5
2
C25 H18 N O
348.1382
0.4092
1.1754
17.5

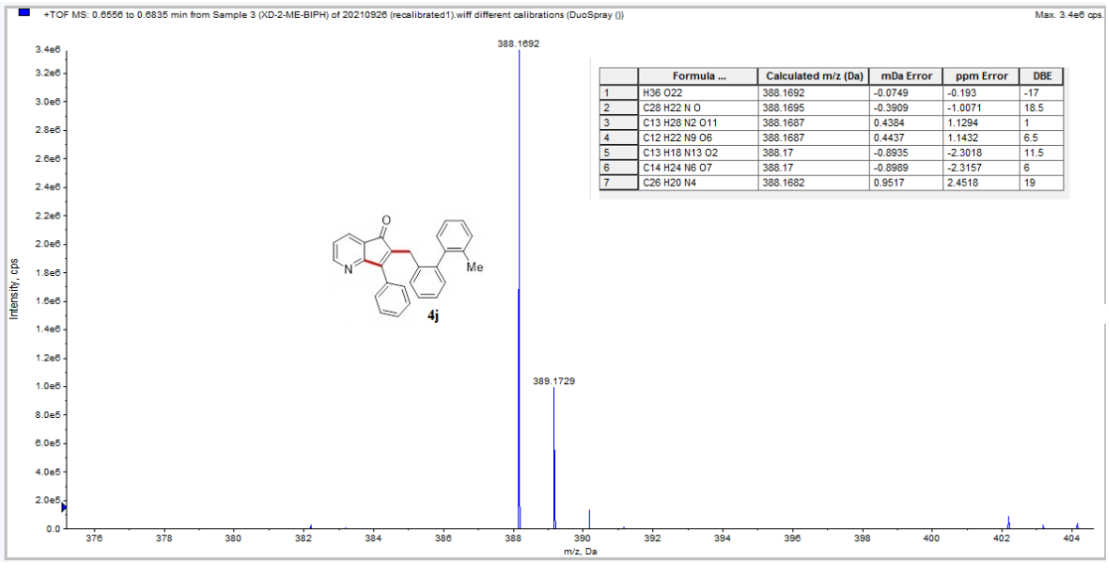

8.4984
8.4958
8.4896
8.4870
7.7184
7.7158
7.7065
7.7040
7.6880
7.6850
7.6746
7.6722
7.4546
7.4445
7.4316
7.4208
7.2690
7.1565
7.1419
7.1299
7.1210
7.0781
7.0655
6.8514
6.8378
6.8133
6.8010
6.7877
4.5559
4.5458
4.5357
4.5256
3.8500
1.2593
1.2493
4i
CDCl3, 600 MHz
H2O
1.00
1.09
2.05
3.07
2.02
1.00
1.04
1.06
1.23
2.14
3.33
3.44
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

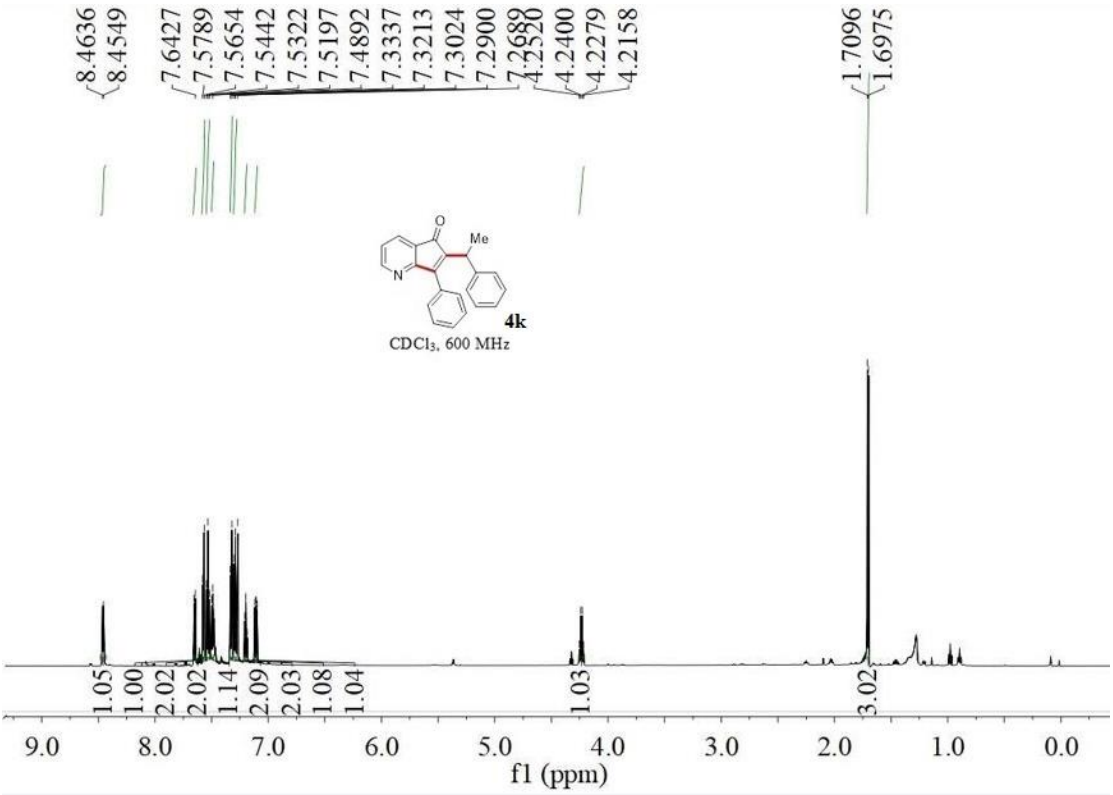

195.8789
167.4623
155.5324
151.7591
129.8311
129.3992
129.1847
128.9441
128.5277
128.4424
128.0449
127.3690
125.5642
122.1937
120.2453
112.7773
77.0719
24.1399
22.1655
4i
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

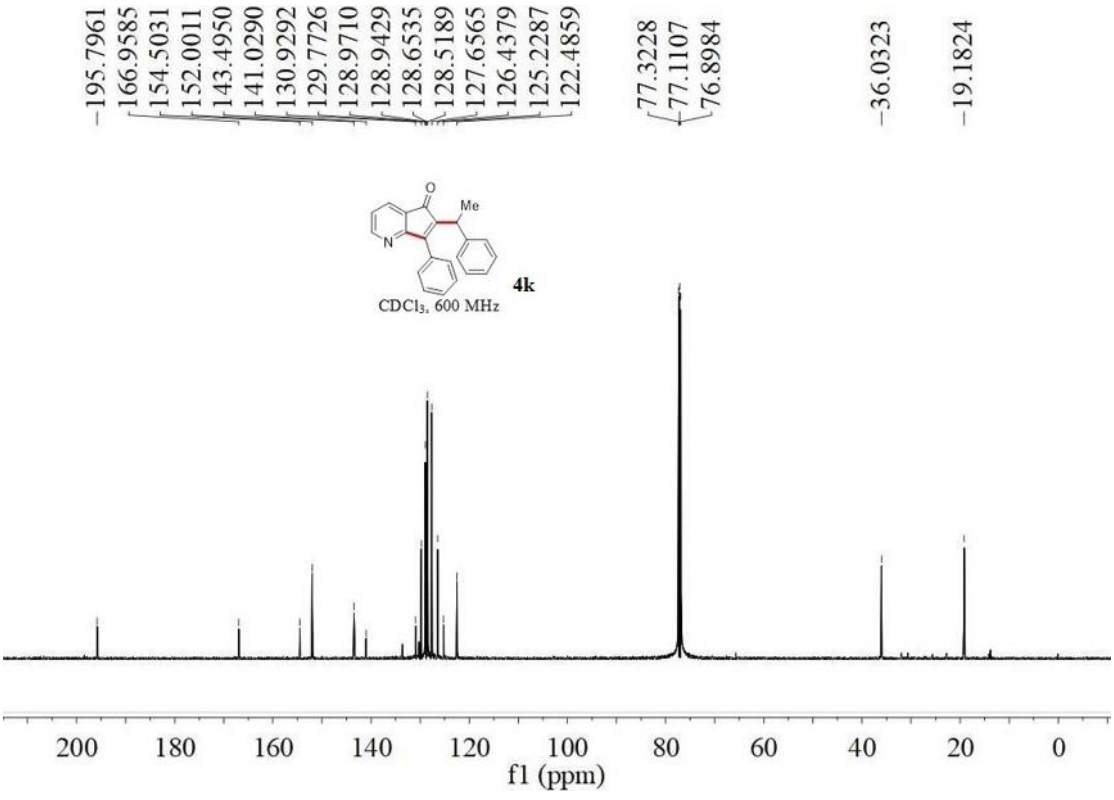

+TOF MS: 0.5905 to 0.6231 min from Sample 17 (XD-2-YIBINYANG-TOWENE) of 20210928 (recalibrated).wiff different calibrations (DuoSpray ())
Max. 9.4e5 cps.
356.1648
357.1689
Intensity, cps
m/z, Da
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C10 H24 N6 O8 | 356.165 | -0.2135 | -0.5995 | 2
2 | C24 H22 N O2 | 356.1645 | 0.2944 | 0.8266 | 14.5
4i

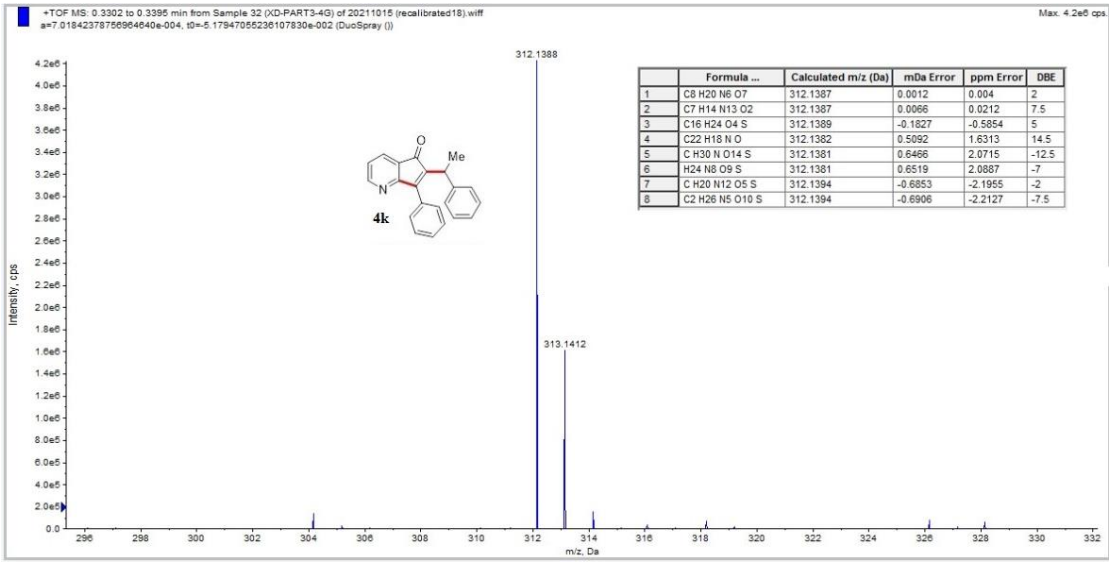

8.4985
8.4958
8.4897
8.4871
7.6854
7.6828
7.6734
7.6708
7.6233
7.6173
7.6111
7.6073
7.4401
7.4337
7.4292
7.2688
7.2611
7.2552
7.2510
7.2288
7.2250
7.2222
7.1940
7.1832
7.1769
7.1710
7.1606
7.1561
7.1416
7.1328
7.1296
7.1208
3.6038
3.5470
2.1108
Me
4j
CDCl3, 600 MHz
1.00
1.05
2.01
3.01
1.15
2.07
0.95
2.10
2.01
1.02
1.02
1.05
3.04
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

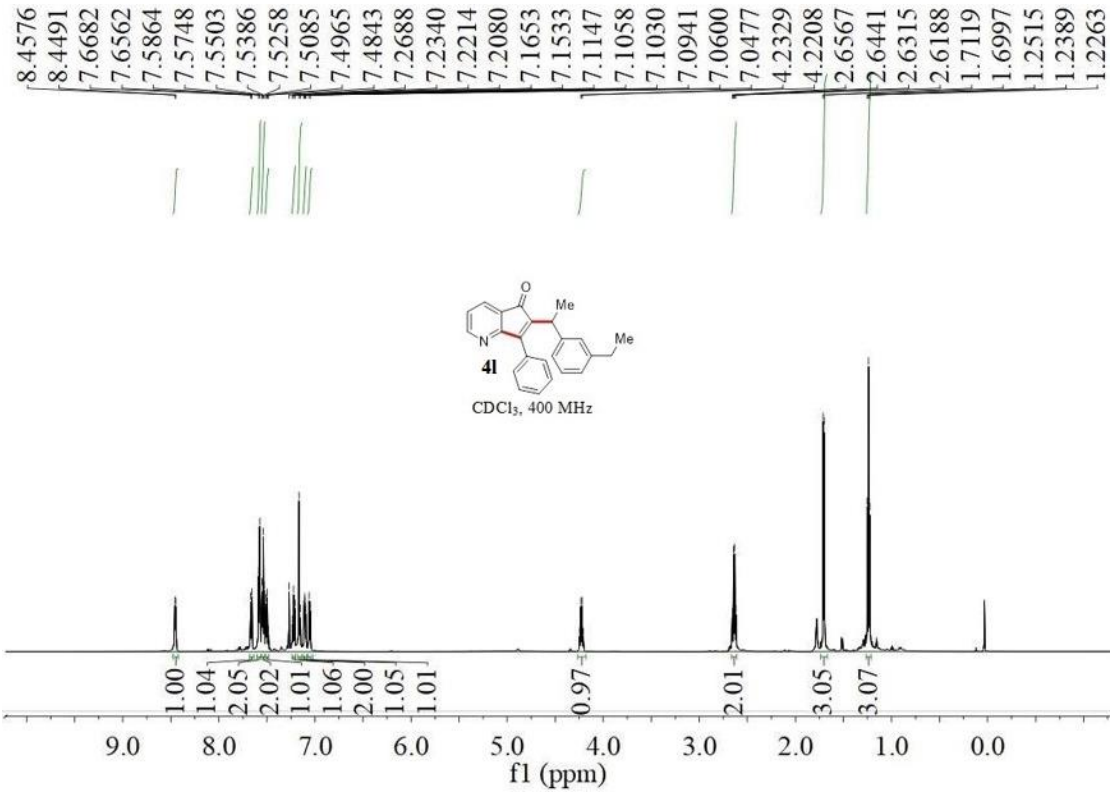

195.3915
151.8199
136.6037
136.0589
130.0931
130.0112
129.8225
129.5457
129.0270
129.0015
128.5114
127.8754
127.5881
127.4692
126.3500
125.6847
125.4010
77.3318
77.1197
76.9073
27.5691
20.0056
O
N
Me
4j
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

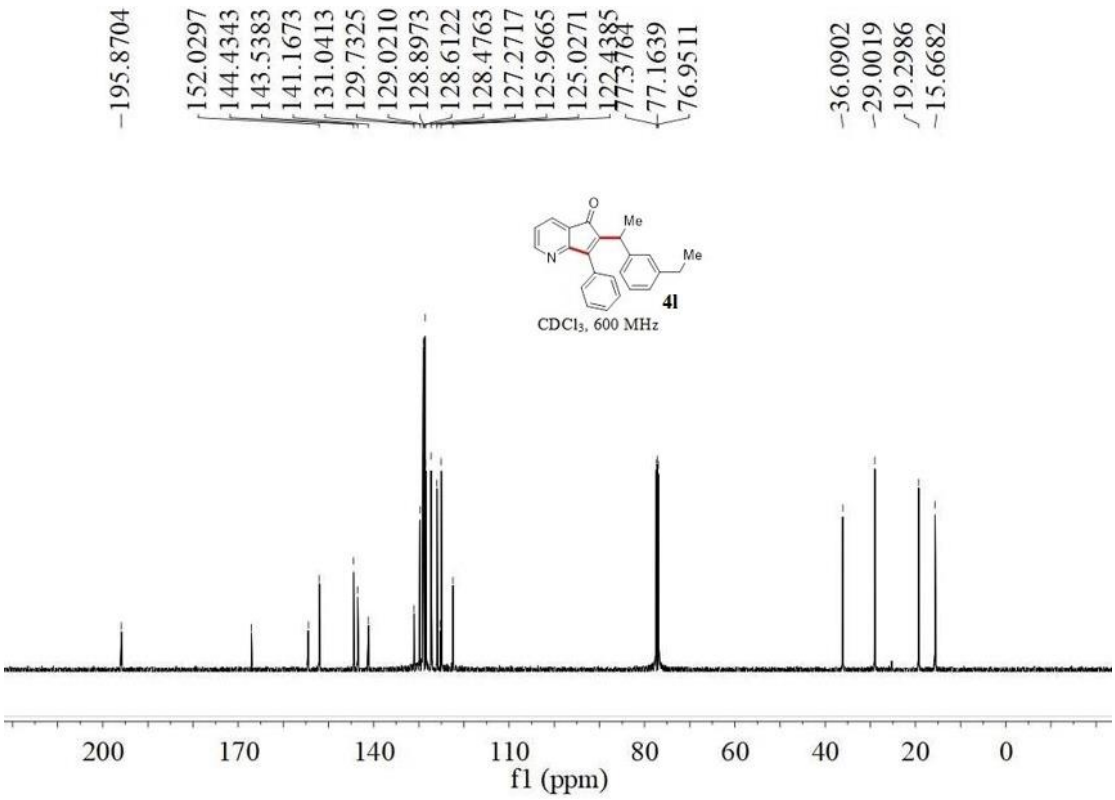

+TOF MS: 0.6556 to 0.6835 min from Sample 3 (XD-2-ME-BIPH) of 20210926 (recalibrated1).wiff different calibrations (DuoSpray ())
Max. 3.4e6 cps.
388.1692
389.1729
Intensity, cps
m/z, Da
O
N
Me
4j
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | H36 O22 | 388.1692 | -0.0749 | -0.193 | -17
2 | C28 H22 N O | 388.1695 | -0.3909 | -1.0071 | 18.5
3 | C13 H28 N2 O11 | 388.1687 | 0.4384 | 1.1294 | 1
4 | C12 H22 N9 O6 | 388.1687 | 0.4437 | 1.1432 | 6.5
5 | C13 H18 N13 O2 | 388.17 | -0.8935 | -2.3018 | 11.5
6 | C14 H24 N6 O7 | 388.17 | -0.8989 | -2.3157 | 6
7 | C26 H20 N4 | 388.1682 | 0.9517 | 2.4518 | 19

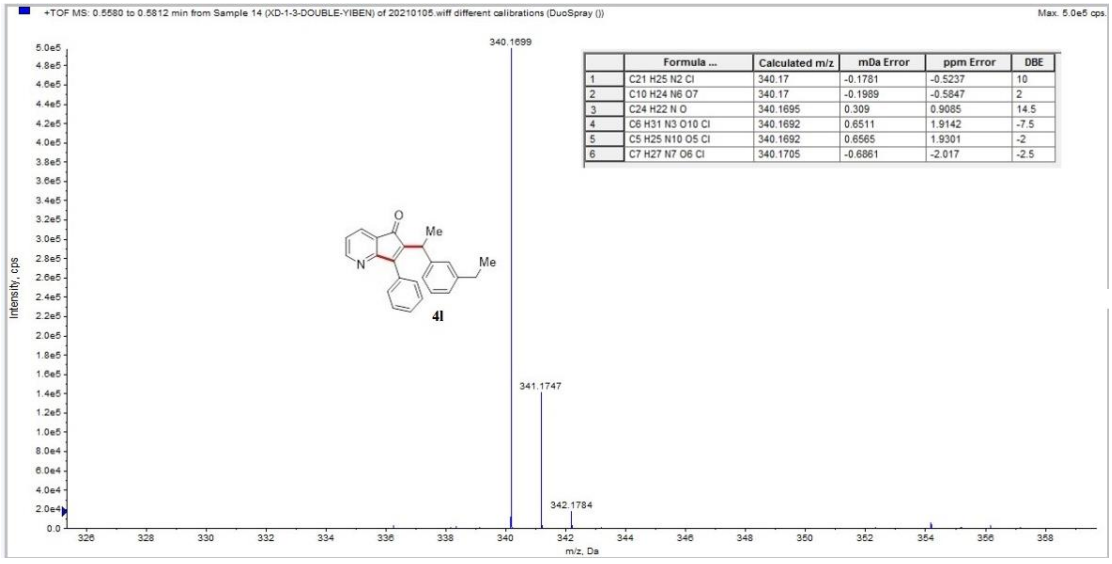

8.4636
8.4549
7.6427
7.5789
7.5654
7.5442
7.5322
7.5197
7.4892
7.3337
7.3213
7.3024
7.2900
4.2400
4.2279
4.2158
1.7096
1.6975
Me
4k
CDCl3, 600 MHz
1.05
1.00
2.02
2.02
1.14
2.09
2.03
1.08
1.04
1.03
3.02
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)


195.7961
166.9585
154.5031
152.0011
143.4950
141.0290
130.9292
129.7726
128.9710
128.9429
128.6535
128.5189
127.6565
126.4379
125.2287
122.4859
77.3228
77.1107
76.8984
36.0323
19.1824
Me
4k
CDCl3, 600 MHz
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

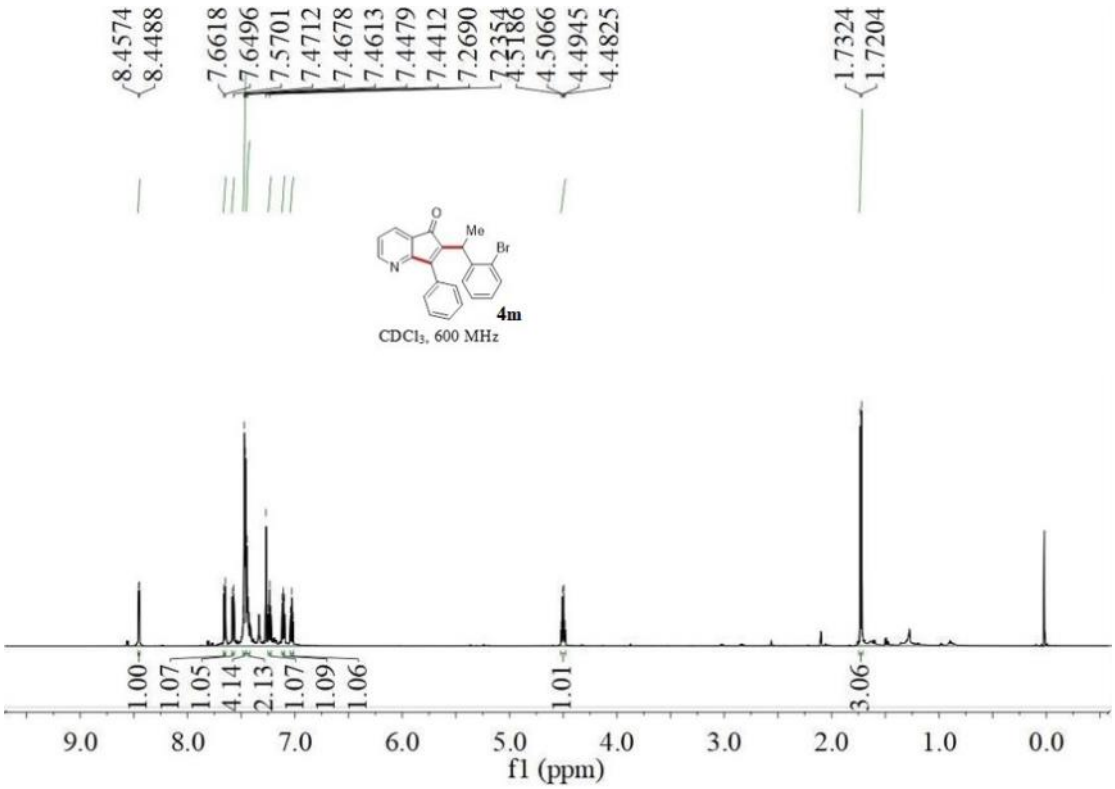

+TOF MS: 0.3302 to 0.3395 min from Sample 32 (XD-PART3-4G) of 20211015 (recalibrated18).wiff
a=7.0184237875696464 0e-004, t0=-5.17947055236107830e-002 (DuoSpray ())
Max. 4.2e6 cps.
312.1388
313.1412
Intensity, cps
m/z, Da
4k
Me
O
N
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C8 H20 N6 O7 | 312.1387 | 0.0012 | 0.004 | 2
2 | C7 H14 N13 O2 | 312.1387 | 0.0066 | 0.0212 | 7.5
3 | C16 H24 O4 S | 312.1389 | -0.1827 | -0.5854 | 5
4 | C22 H18 N O | 312.1382 | 0.5092 | 1.6313 | 14.5
5 | C H30 N O14 S | 312.1381 | 0.6466 | 2.0715 | -12.5
6 | H24 N8 O9 S | 312.1381 | 0.6519 | 2.0887 | -7
7 | C H20 N12 O5 S | 312.1394 | -0.6853 | -2.1955 | -2
8 | C2 H26 N5 O10 S | 312.1394 | -0.6906 | -2.2127 | -7.5

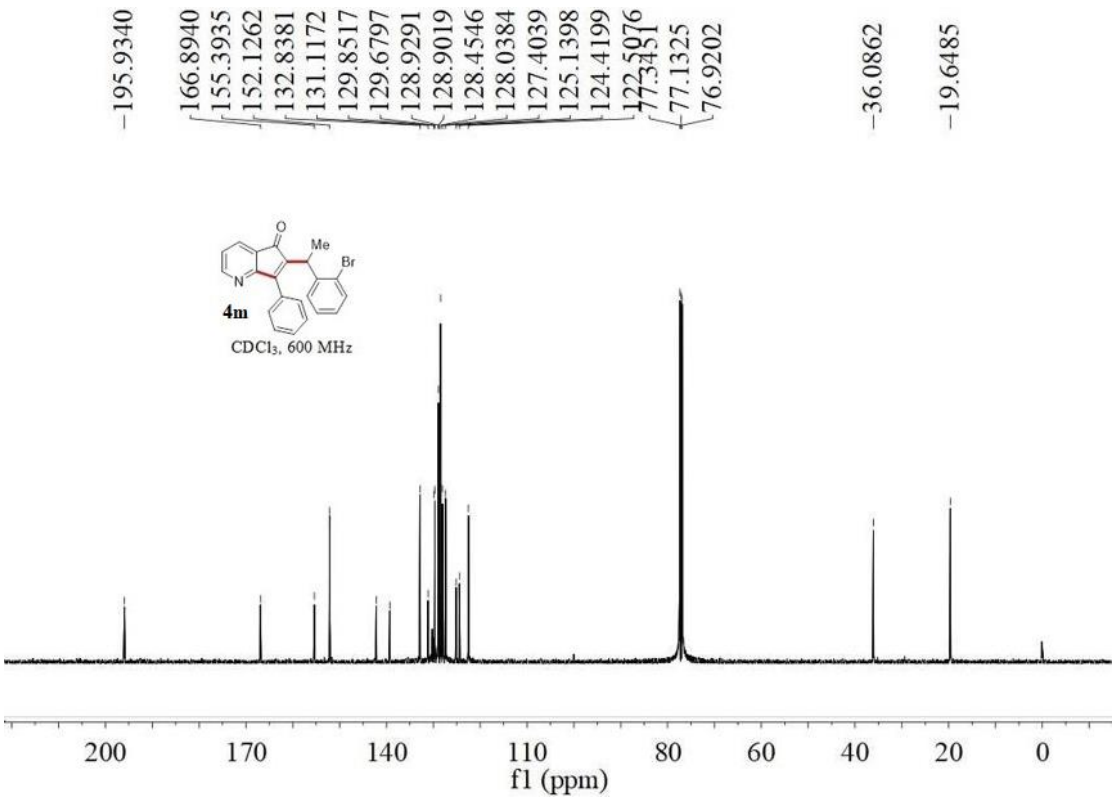

8.4576
8.4491
7.6682
7.6562
7.5864
7.5748
7.5503
7.5386
7.5258
7.5085
7.4965
7.4843
7.2688
7.2340
7.2214
7.2080
7.1653
7.1533
7.1147
7.1058
7.1030
7.0941
7.0600
7.0477
4.2329
4.2208
2.6567
2.6441
2.6315
2.6188
1.7119
1.6997
1.2515
1.2389
1.2263
4l
Me
Me
O
N
CDCl3, 400 MHz
1.00
1.04
2.05
2.02
1.01
1.06
2.00
1.05
1.01
0.97
2.01
3.05
3.07
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

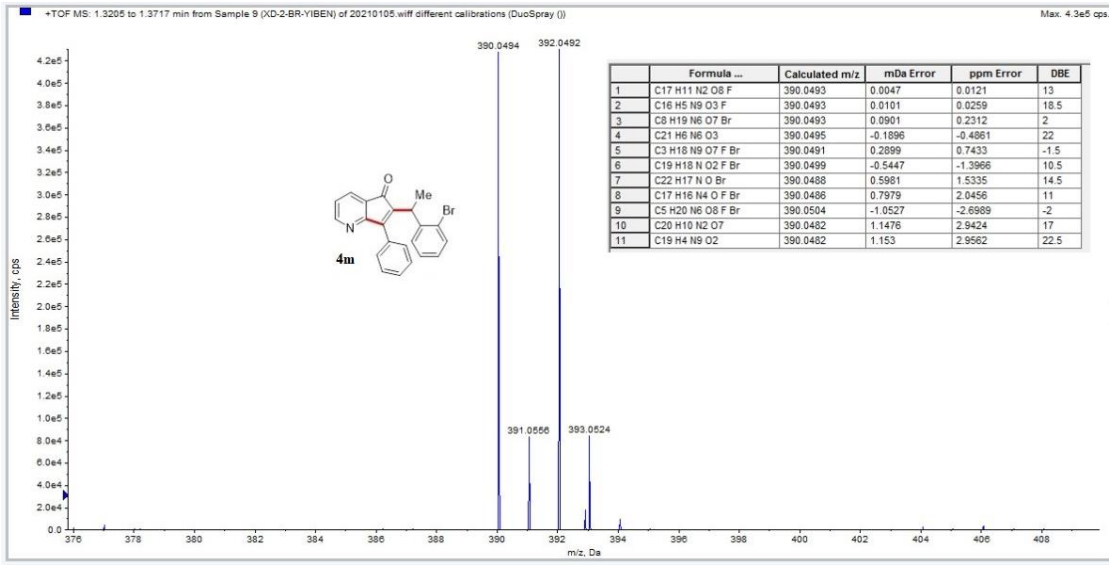




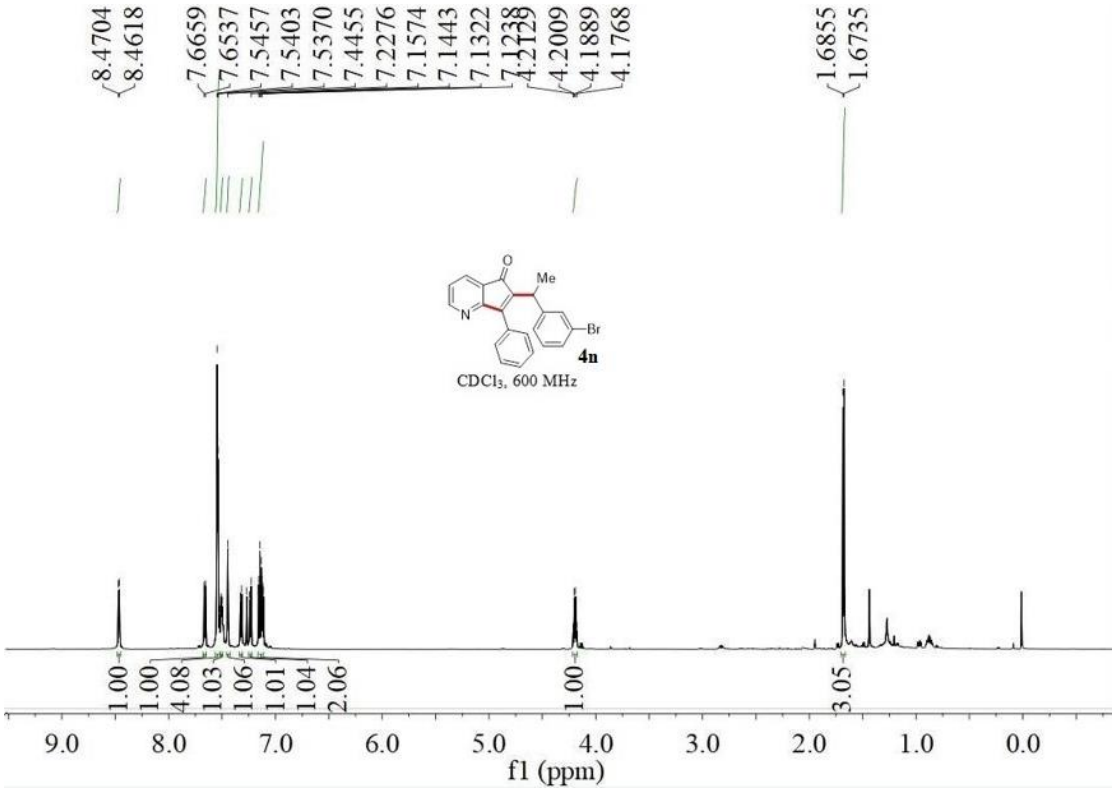
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C21 H25 N2 Cl	340.17	-0.1781	-0.5237	10
2	C10 H24 N6 O7	340.17	-0.1989	-0.5847	2
3	C24 H22 N O	340.1695	0.309	0.9085	14.5
4	C6 H31 N3 O10 Cl	340.1692	0.6511	1.9142	-7.5
5	C5 H25 N10 O5 Cl	340.1692	0.6565	1.9301	-2
6	C7 H27 N7 O6 Cl	340.1705	-0.6861	-2.017	-2.5

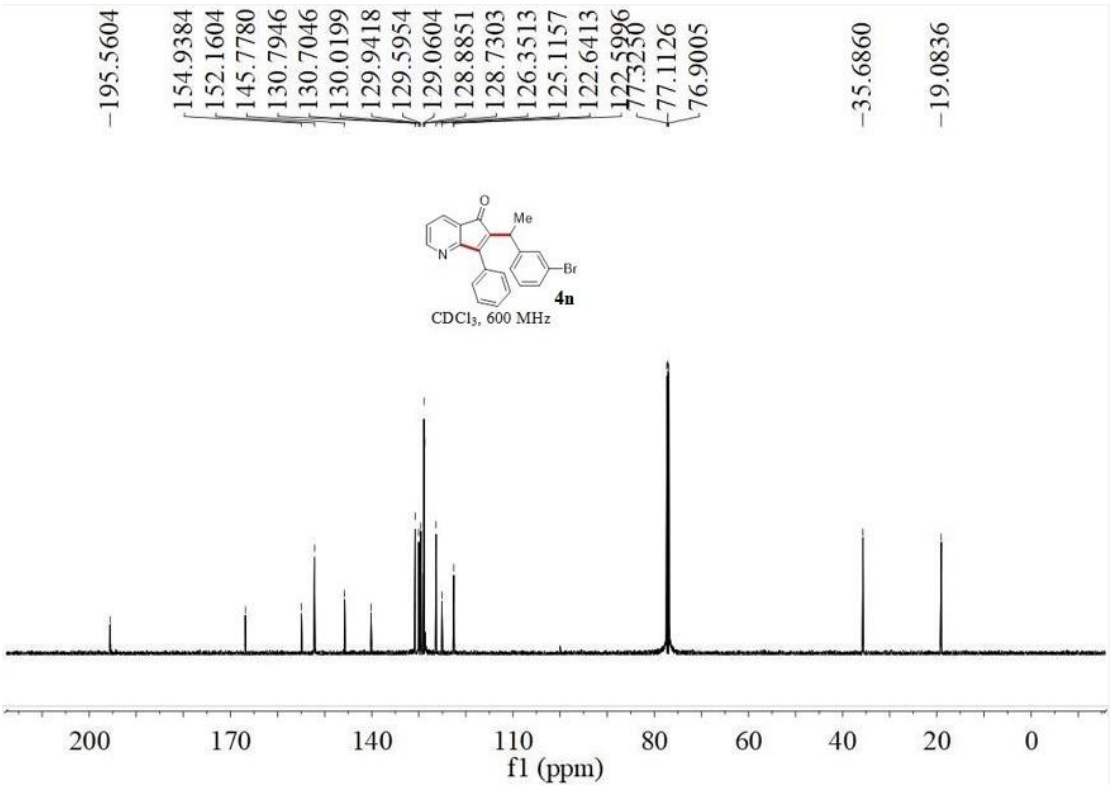

8.4574
8.4488
7.6618
7.6496
7.5701
7.4712
7.4678
7.4613
7.4479
7.4412
7.2690
4.5066
4.4945
4.4825
1.7324
1.7204
Me
Br
O
N
4m
CDCl3, 600 MHz
1.00
1.07
1.05
4.14
2.13
1.07
1.09
1.06
1.01
3.06
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

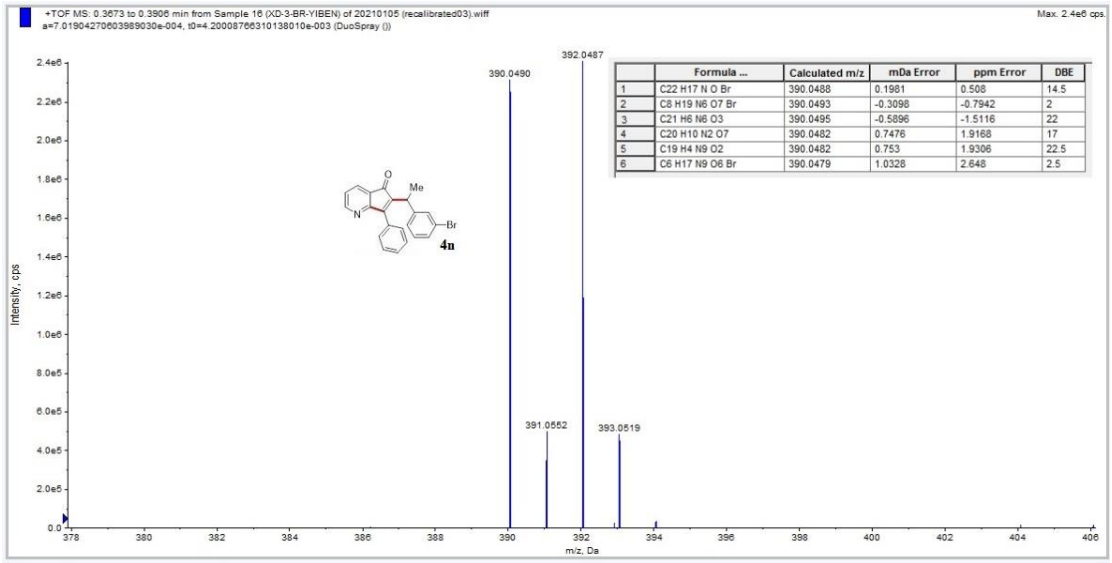

195.9340
166.8940
155.3935
152.1262
132.8381
131.1172
129.8517
129.6797
128.9291
128.9019
128.4546
128.0384
127.4039
125.1398
124.4199
77.1325
76.9202
36.0862
19.6485
Me
Br
O
N
4m
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

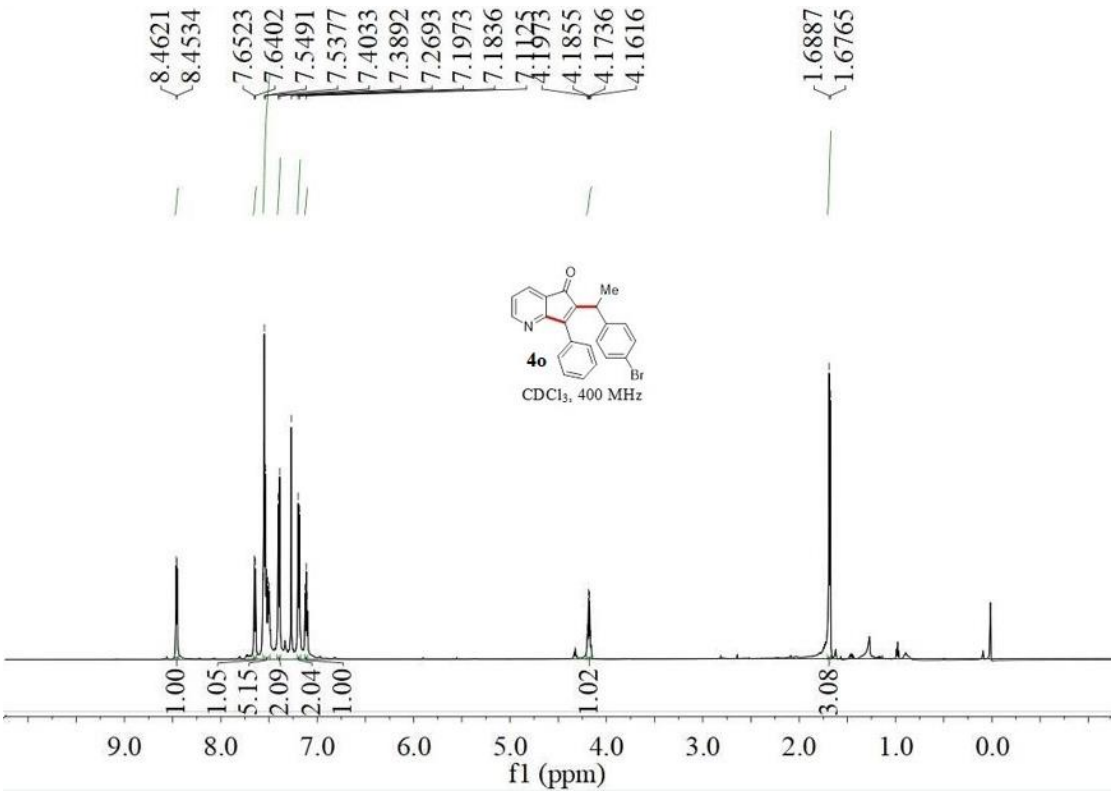


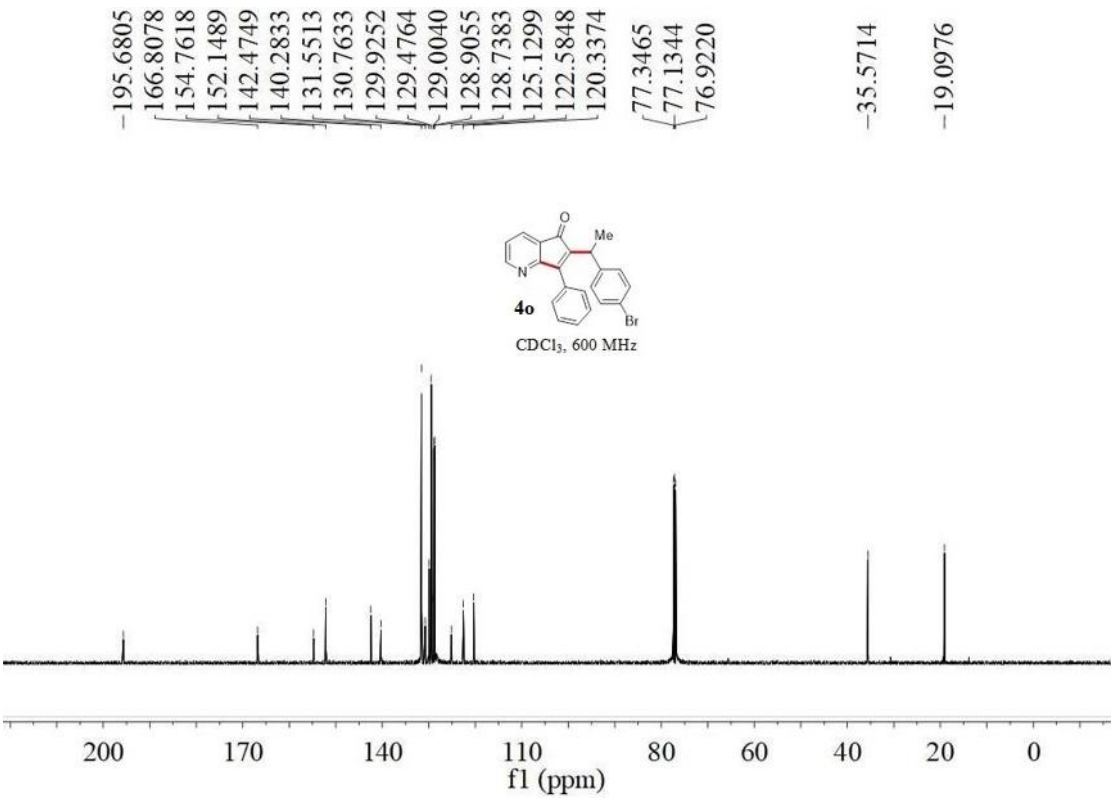
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C17 H11 N2 O8 F	390.0493	0.0047	0.0121	13
2	C16 H5 N9 O3 F	390.0493	0.0101	0.0259	18.5
3	C8 H19 N6 O7 Br	390.0493	0.0901	0.2312	2
4	C21 H6 N6 O3	390.0495	-0.1896	-0.4861	22
5	C3 H18 N9 O7 F Br	390.0491	0.2899	0.7433	-1.5
6	C19 H18 N O2 F Br	390.0499	-0.5447	-1.3966	10.5
7	C22 H17 N O Br	390.0488	0.5981	1.5335	14.5
8	C17 H16 N4 O F Br	390.0486	0.7979	2.0456	11
9	C5 H20 N6 O8 F Br	390.0504	-1.0527	-2.6989	-2
10	C20 H10 N2 O7	390.0482	1.1476	2.9424	17
11	C19 H4 N9 O2	390.0482	1.153	2.9562	22.5

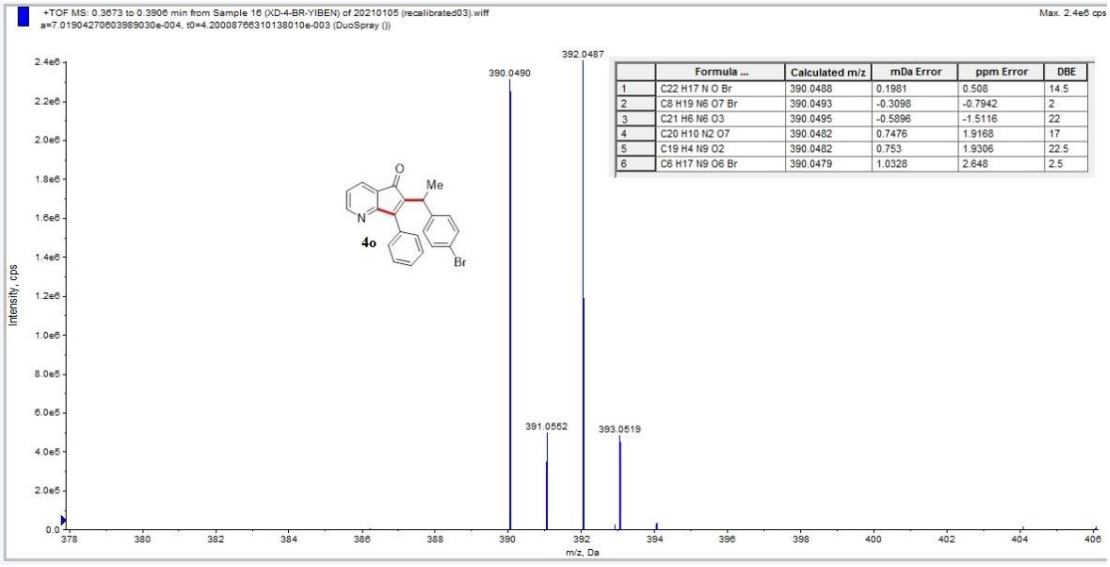



195.5604
154.9384
152.1604
145.7780
130.7946
130.7046
130.0199
129.9418
129.5954
129.0604
128.8851
128.7303
126.3513
125.1157
122.6413
122.5996
77.3250
77.1126
76.9005
35.6860
19.0836
O
Me
N
Br
4n
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

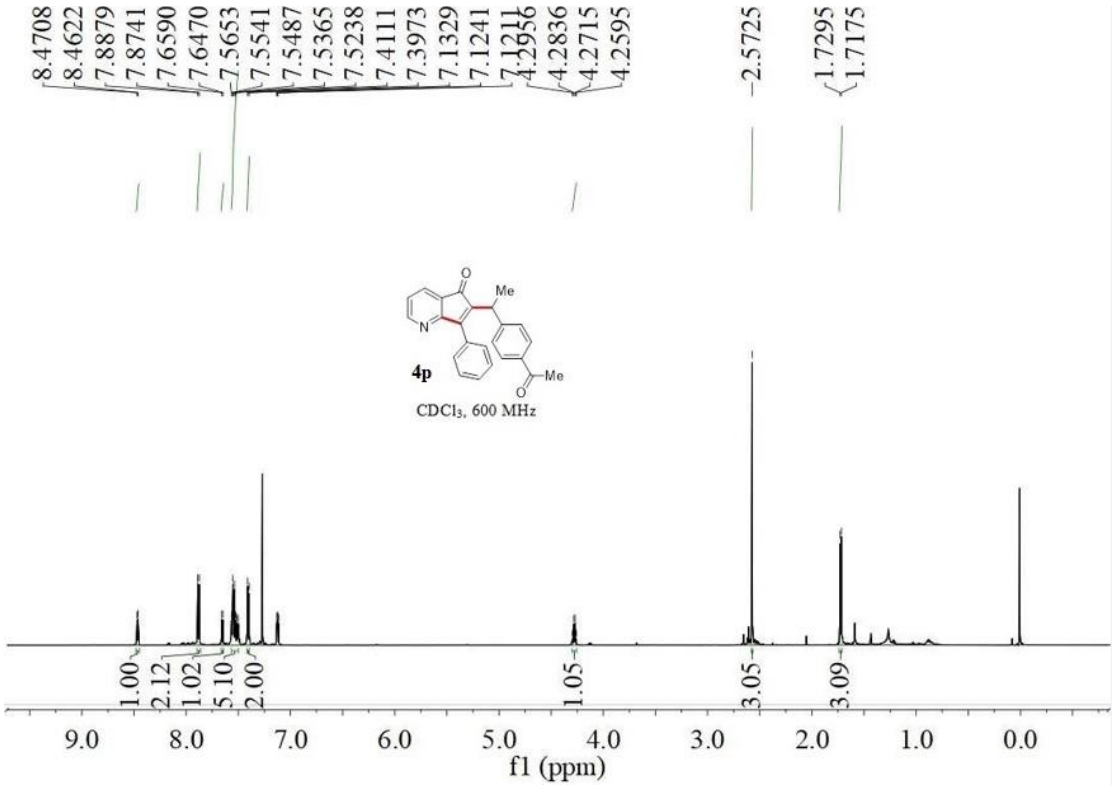

+TOF MS: 0.3673 to 0.3906 min from Sample 16 (XD-3-BR-YIBEN) of 20210105 (recalibrated03).wiff
a=7.0190427660398903e-004, t0=4.2000876631013801e-003 (DuoSpray ())
Max. 2.4e6 cps.
392.0487
390.0490
391.0552
393.0519
Intensity, cps
m/z, Da
4n
Formula ... | Calculated m/z | mDa Error | ppm Error | DBE
1 | C22 H17 N O Br | 390.0488 | 0.1981 | 0.508 | 14.5
2 | C8 H19 N6 O7 Br | 390.0493 | -0.3098 | -0.7942 | 2
3 | C21 H6 N6 O3 | 390.0495 | -0.5896 | -1.5116 | 22
4 | C20 H10 N2 O7 | 390.0482 | 0.7476 | 1.9168 | 17
5 | C19 H4 N9 O2 | 390.0482 | 0.753 | 1.9306 | 22.5
6 | C6 H17 N9 O6 Br | 390.0479 | 1.0328 | 2.648 | 2.5

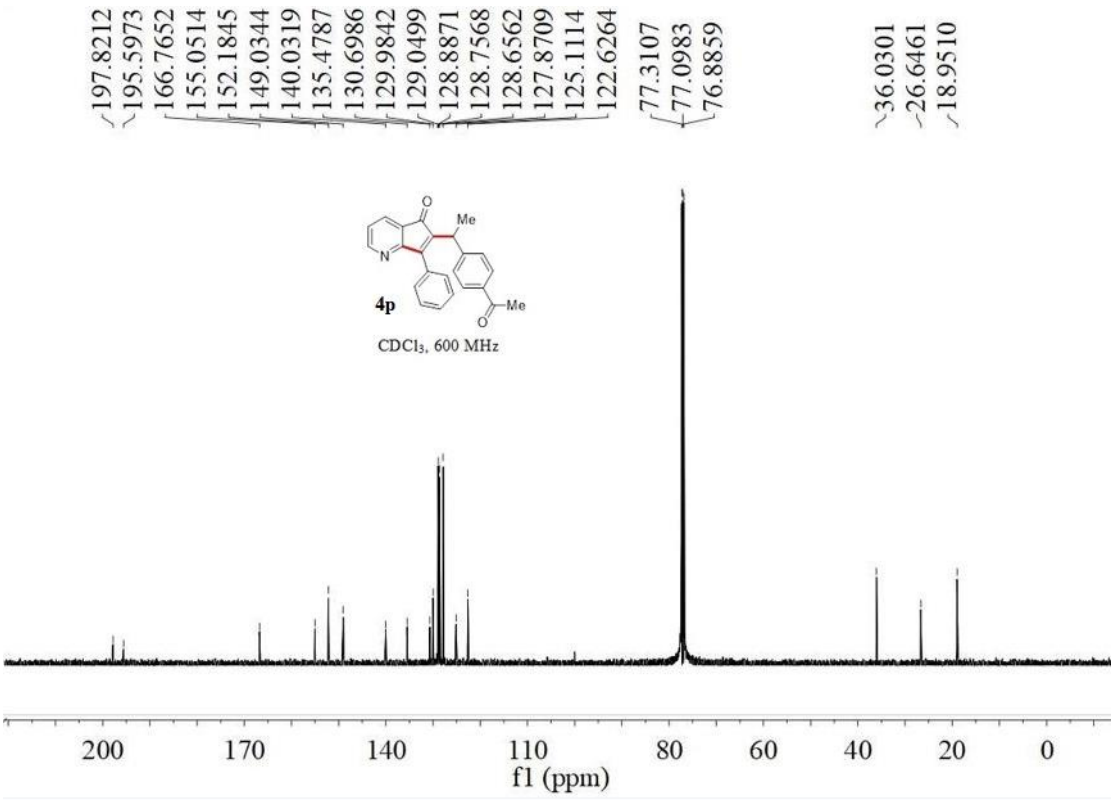

8.4621
8.4534
7.6523
7.6402
7.5491
7.5377
7.4033
7.3892
7.2693
7.1973
7.1836
7.1125
4.1973
4.1855
4.1736
4.1616
1.6887
1.6765
O
Me
N
4o
Br
CDCl3, 400 MHz
1.00
1.05
5.15
2.09
2.04
1.00
1.02
3.08
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

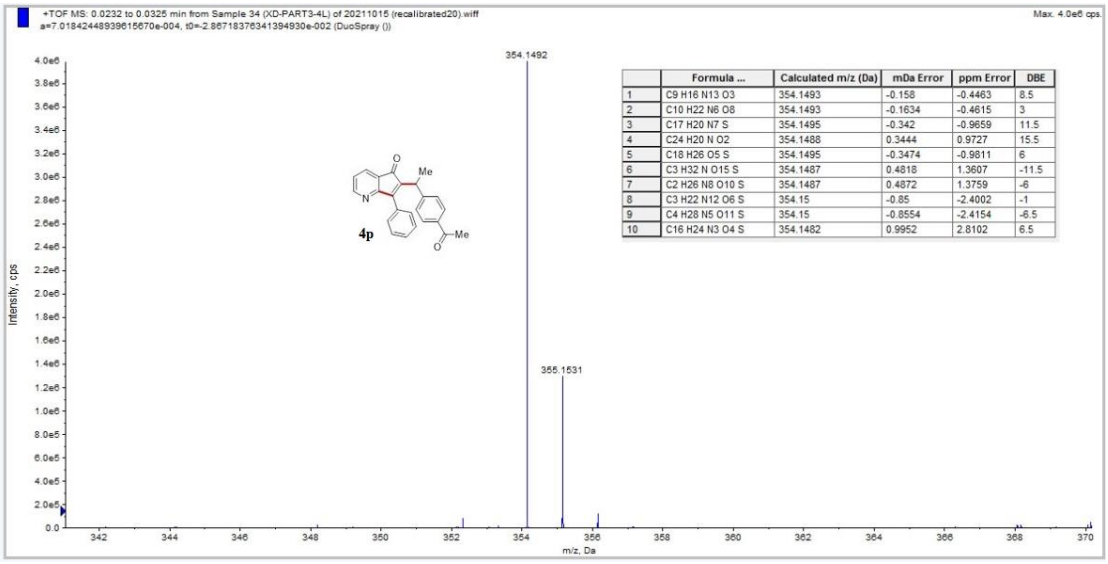

195.6805
166.8078
154.7618
152.1489
142.4749
140.2833
131.5513
130.7633
129.9252
129.4764
129.0040
128.9055
128.7383
125.1299
122.5848
120.3374
77.3465
77.1344
76.9220
35.5714
19.0976
O
Me
N
4o
Br
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

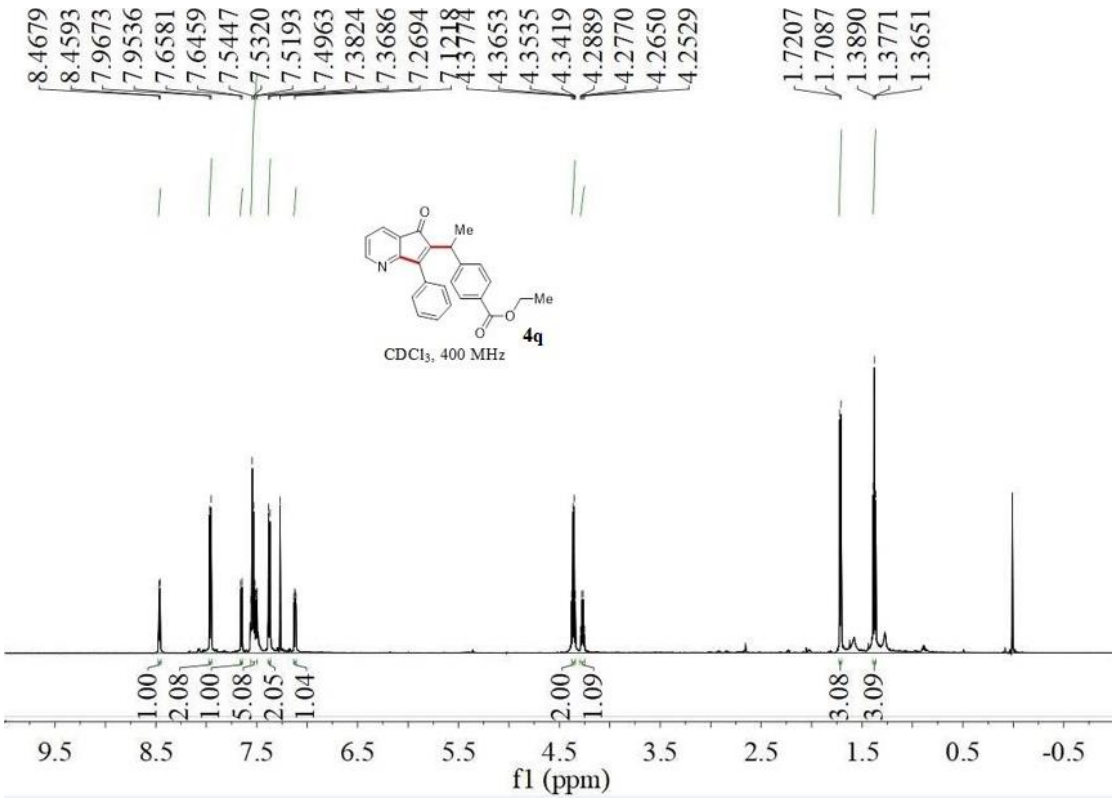


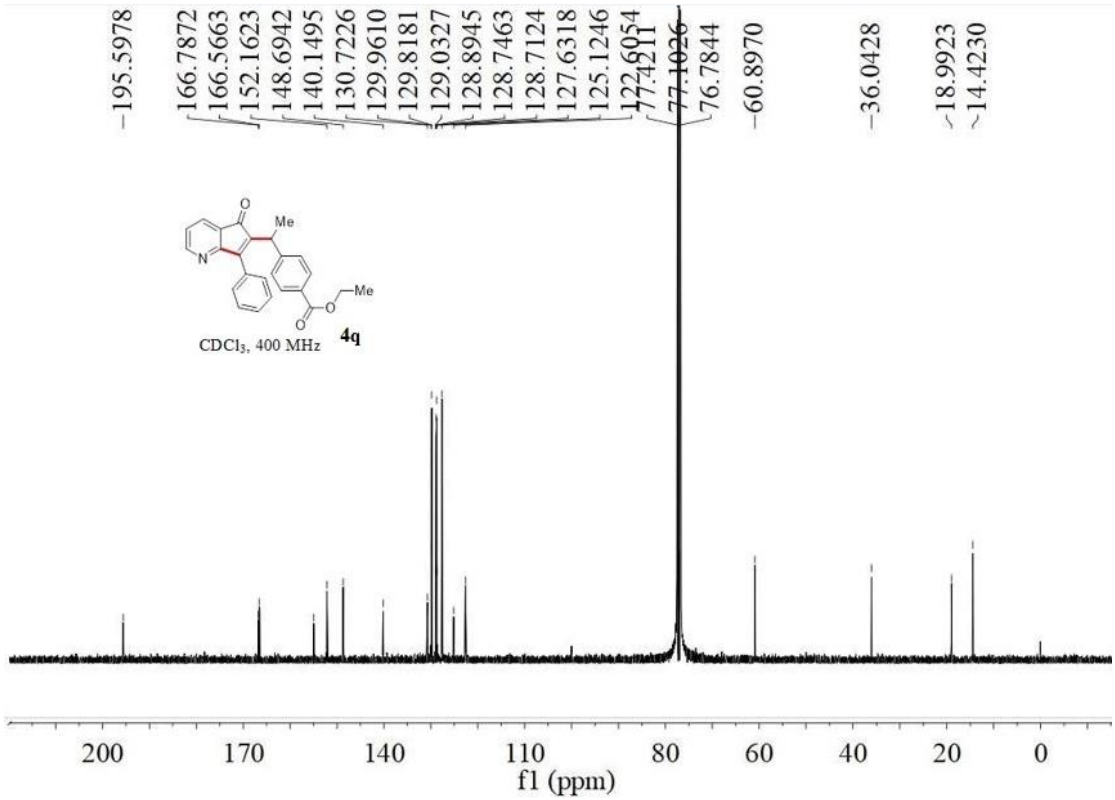
	Formula ...	Calculated m/z	mDa Error	ppm Error	DBE
1	C22 H17 N O Br	390.0488	0.1981	0.508	14.5
2	C8 H19 N6 O7 Br	390.0493	-0.3098	-0.7942	2
3	C21 H6 N6 O3	390.0495	-0.5896	-1.5116	22
4	C20 H10 N2 O7	390.0482	0.7476	1.9168	17
5	C19 H4 N9 O2	390.0482	0.753	1.9306	22.5
6	C6 H17 N9 O6 Br	390.0479	1.0328	2.648	2.5

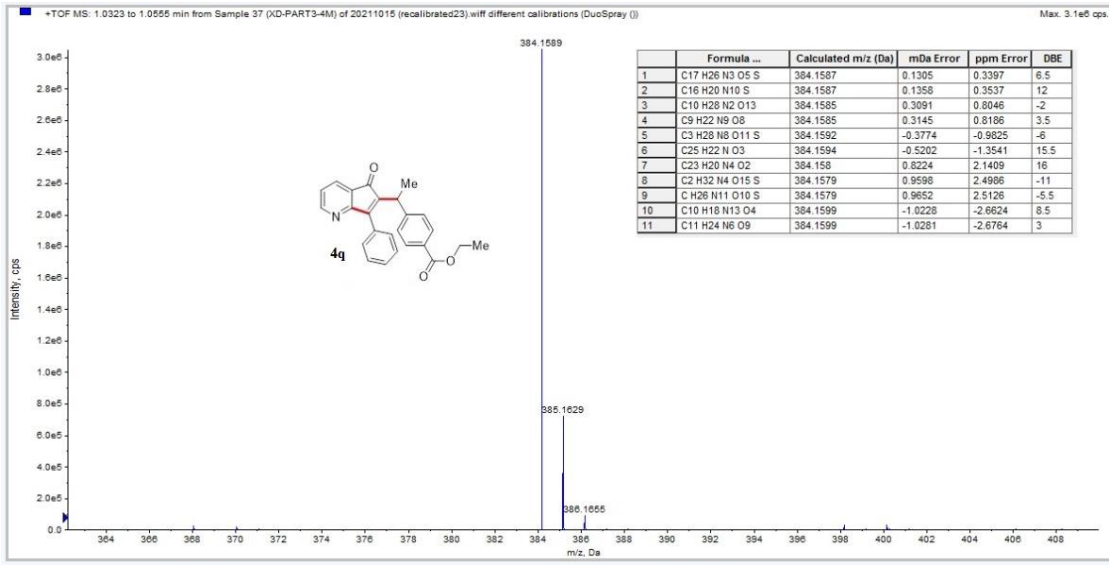



197.8212
195.5973
166.7652
155.0514
152.1845
149.0344
140.0319
135.4787
130.6986
129.9842
129.0499
128.8871
128.7568
128.6562
127.8709
125.1114
122.6264
77.3107
77.0983
76.8859
36.0301
26.6461
18.9510
Me
O
N
4p
Me
O
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

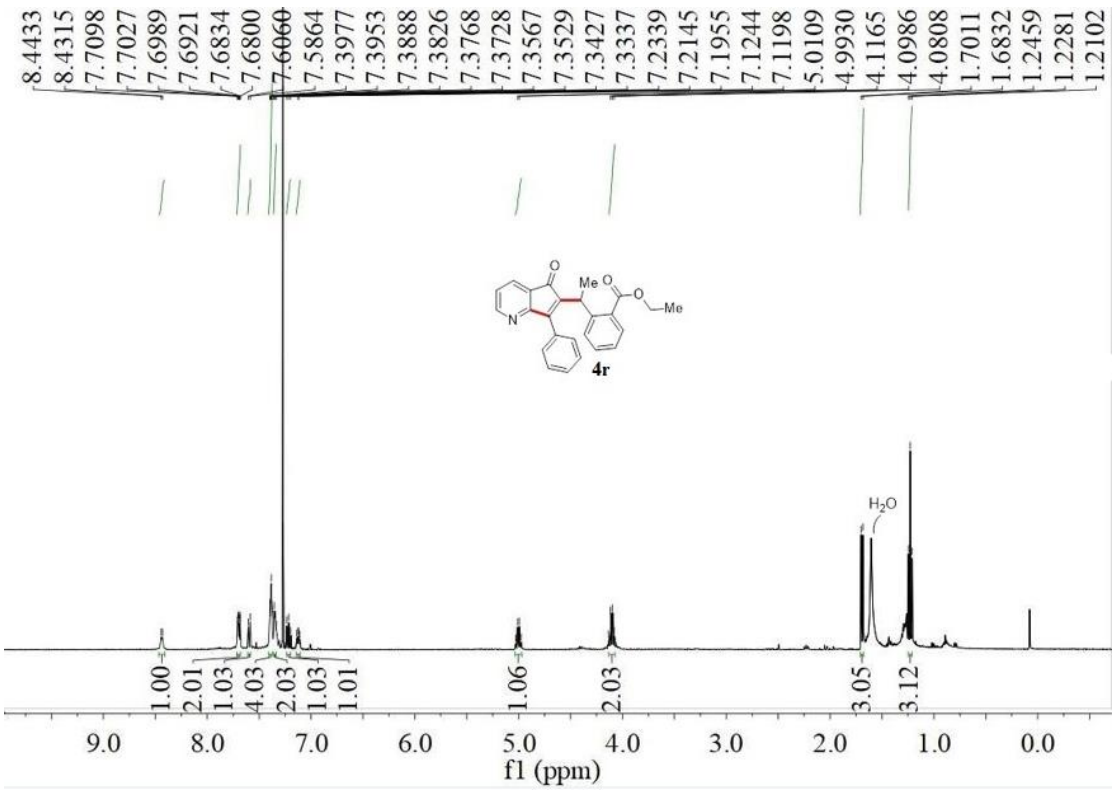

+TOF MS: 0.0232 to 0.0325 min from Sample 34 (XD-PART3-4L) of 20211015 (recalibrated20).wiff
a=7.018424489396156 70e-004, t0=-2.867183768341394930e-002 (DuoSpray ())
Max. 4.0e6 cps.
354.1492
355.1531
Intensity, cps
m/z, Da
4p
Me
O
N
Me
O
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C9 H16 N13 O3 | 354.1493 | -0.158 | -0.4463 | 8.5
2 | C10 H22 N6 O8 | 354.1493 | -0.1634 | -0.4615 | 3
3 | C17 H20 N7 S | 354.1495 | -0.342 | -0.9659 | 11.5
4 | C24 H20 N O2 | 354.1488 | 0.3444 | 0.9727 | 15.5
5 | C18 H26 O5 S | 354.1495 | -0.3474 | -0.9811 | 6
6 | C3 H32 N O15 S | 354.1487 | 0.4818 | 1.3607 | -11.5
7 | C2 H26 N8 O10 S | 354.1487 | 0.4872 | 1.3759 | -6
8 | C3 H22 N12 O6 S | 354.15 | -0.85 | -2.4002 | -1
9 | C4 H28 N5 O11 S | 354.15 | -0.8554 | -2.4154 | -6.5
10 | C16 H24 N3 O4 S | 354.1482 | 0.9952 | 2.8102 | 6.5

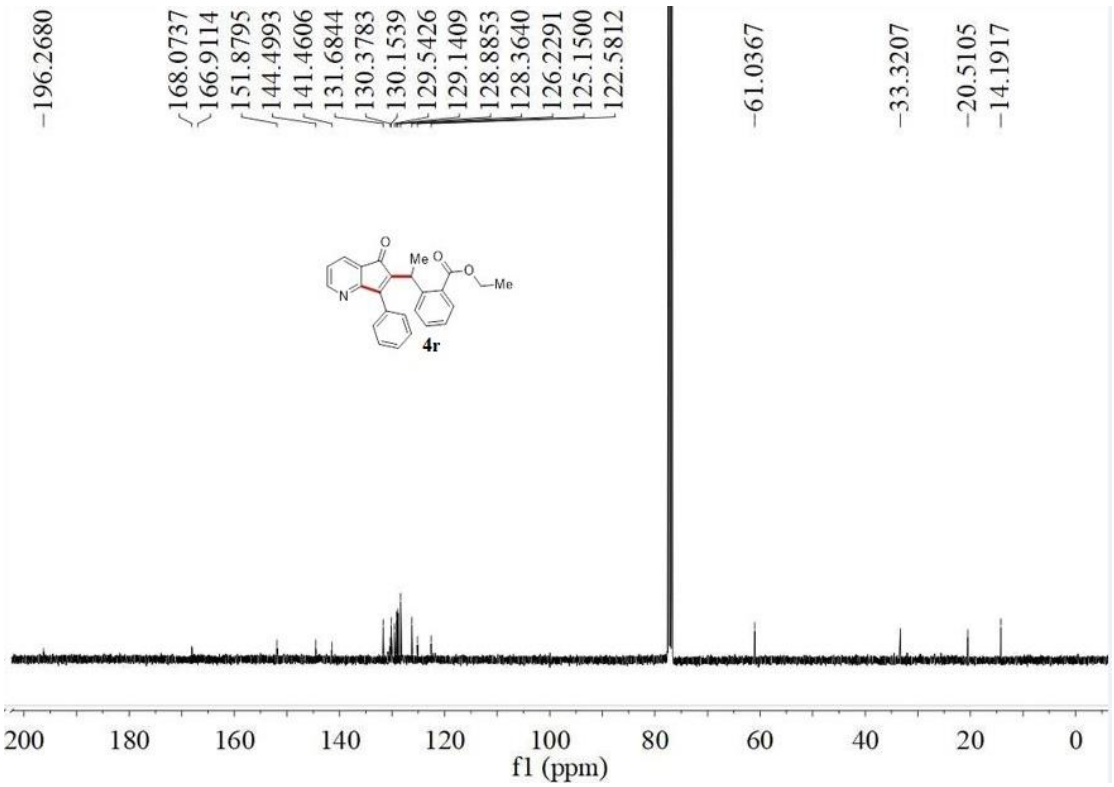

8.4679
8.4593
7.9673
7.9536
7.6581
7.6459
7.5447
7.5320
7.5193
7.4963
7.3824
7.3686
7.2694
4.3653
4.3535
4.3419
4.2889
4.2770
4.2650
4.2529
1.7207
1.7087
1.3890
1.3771
1.3651
Me
Me
4q
CDCl3, 400 MHz
1.00
2.08
1.00
5.08
2.05
1.04
2.00
1.09
3.08
3.09
9.5
8.5
7.5
6.5
5.5
4.5
3.5
2.5
1.5
0.5
-0.5
f1 (ppm)

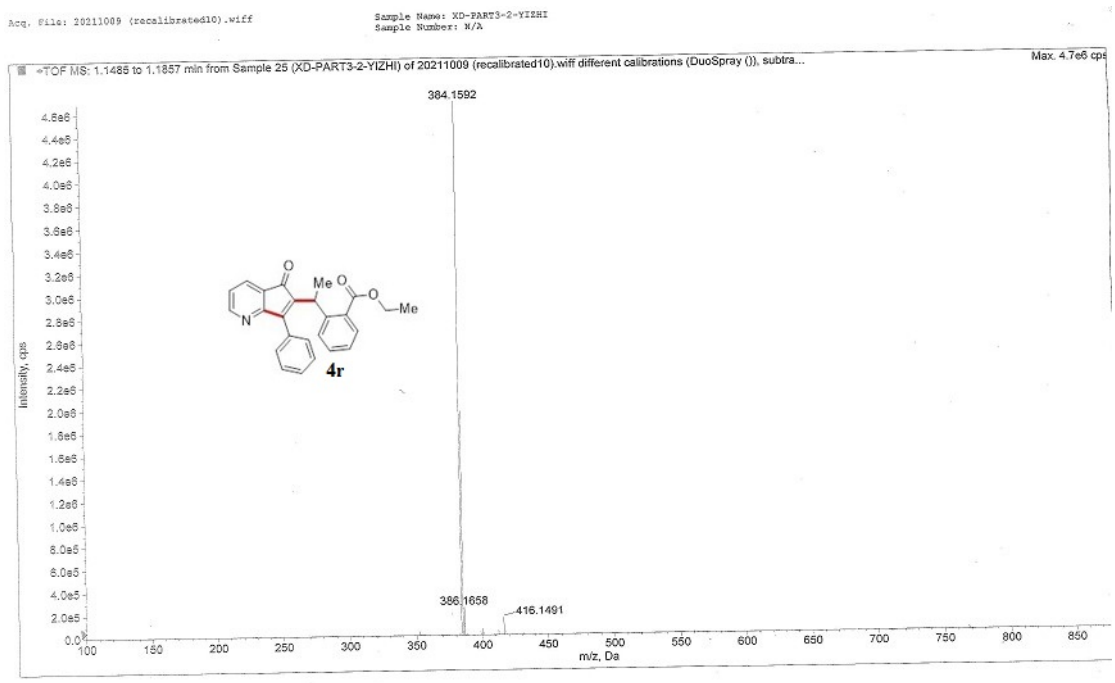

195.5978
166.7872
166.5663
152.1623
148.6942
140.1495
130.7226
129.9610
129.8181
129.0327
128.8945
128.7463
128.7124
127.6318
125.1246
122.6054
77.4211
77.1026
76.7844
60.8970
36.0428
18.9923
14.4230
Me
Me
4q
CDCl3, 400 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

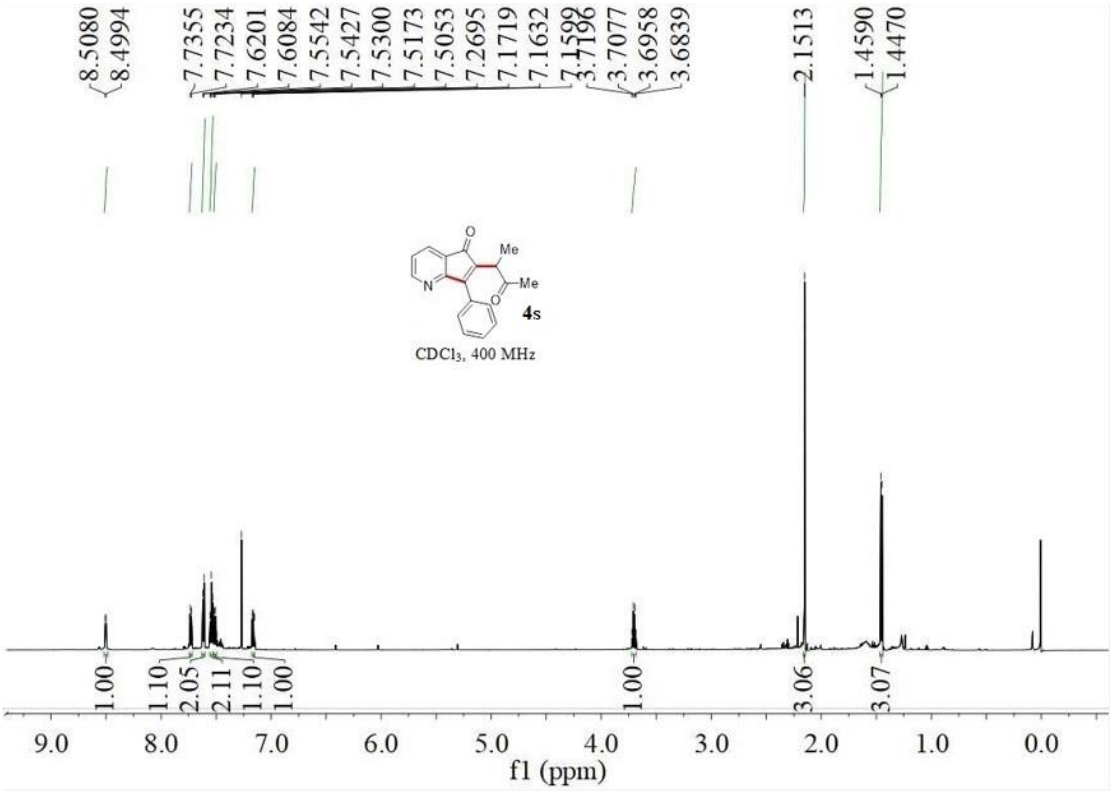


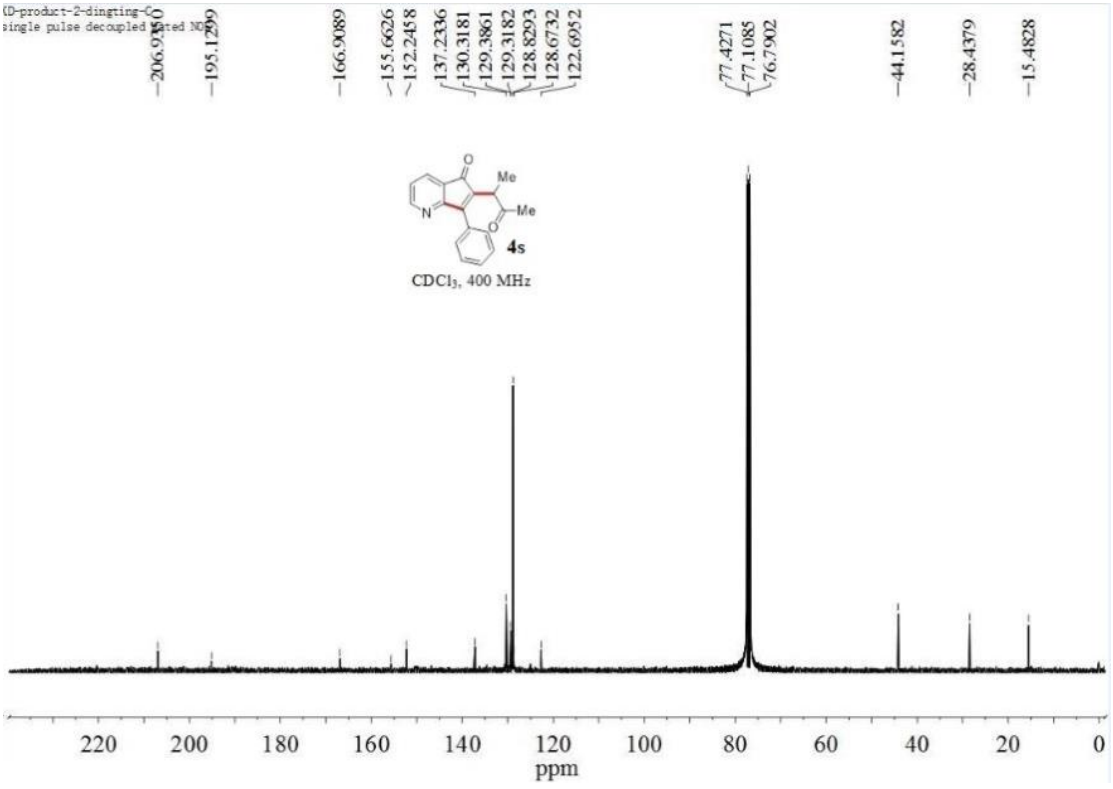
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C17 H26 N3 O5 S	384.1587	0.1305	0.3397	6.5
2	C16 H20 N10 S	384.1587	0.1358	0.3537	12
3	C10 H28 N2 O13	384.1585	0.3091	0.8046	-2
4	C9 H22 N9 O8	384.1585	0.3145	0.8186	3.5
5	C3 H28 N8 O11 S	384.1592	-0.3774	-0.9825	-6
6	C25 H22 N O3	384.1594	-0.5202	-1.3541	15.5
7	C23 H20 N4 O2	384.158	0.8224	2.1409	16
8	C2 H32 N4 O15 S	384.1579	0.9598	2.4986	-11
9	C H26 N11 O10 S	384.1579	0.9652	2.5126	-5.5
10	C10 H18 N13 O4	384.1599	-1.0228	-2.6624	8.5
11	C11 H24 N6 O9	384.1599	-1.0281	-2.6764	3

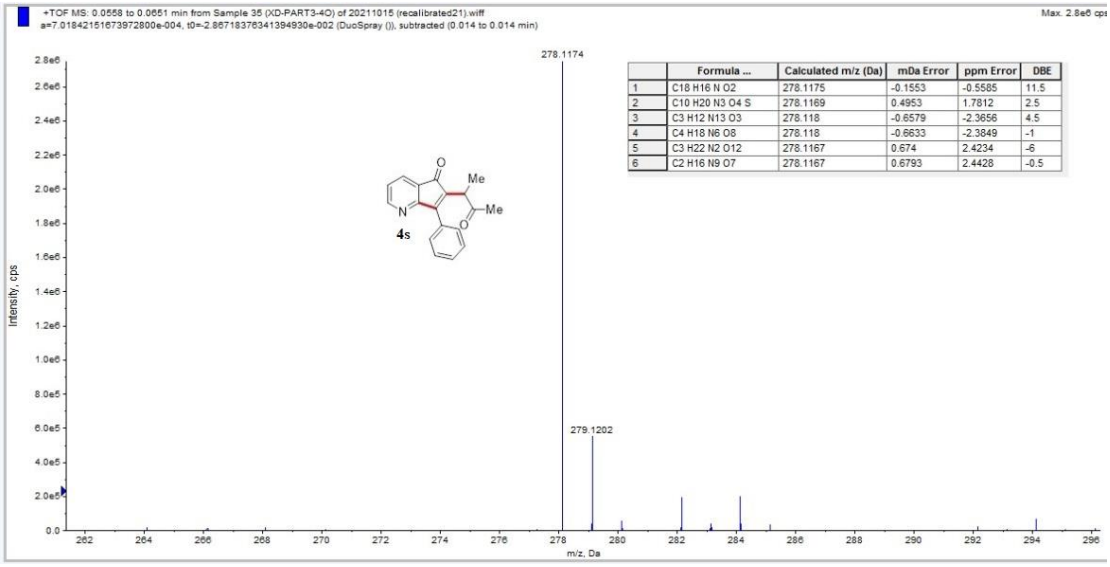



196.2680
168.0737
166.9114
151.8795
144.4993
141.4606
131.6844
130.3783
130.1539
129.5426
129.1409
128.8853
128.3640
126.2291
125.1500
122.5812
61.0367
33.3207
20.5105
14.1917
Me
O
O
Me
N
4r
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)

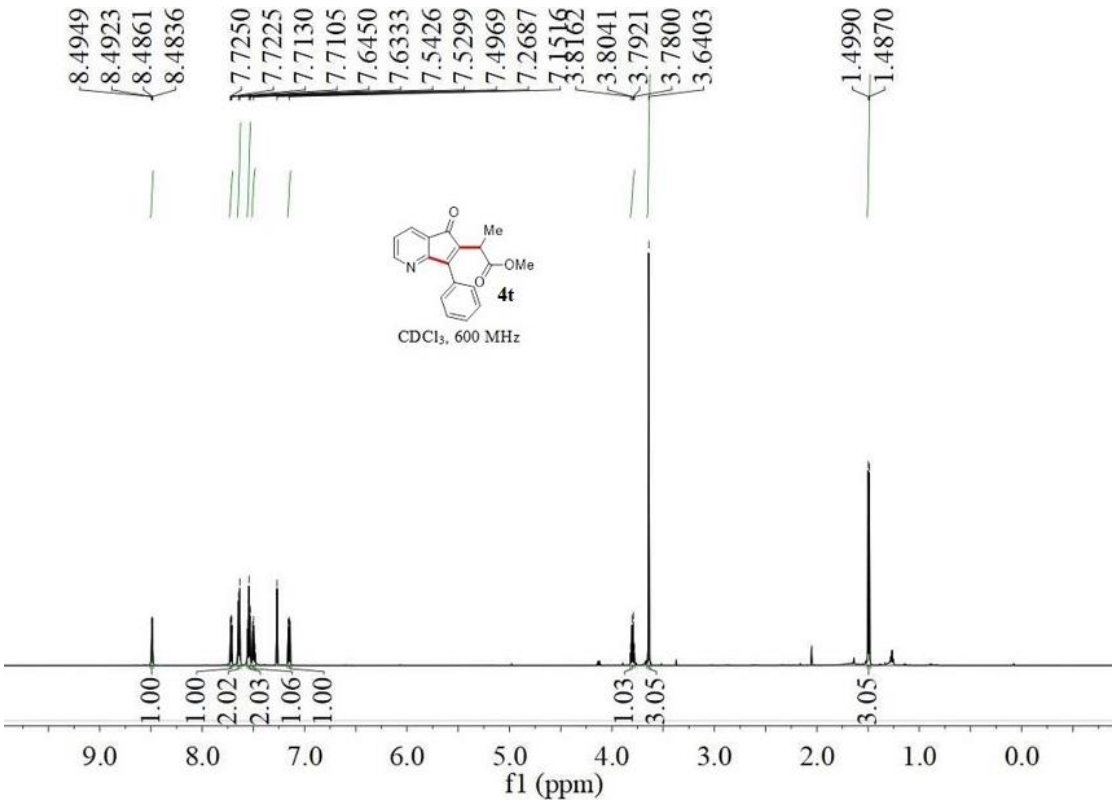

Acq. File: 20211009 (recalibrated10).wiff
Sample Name: XD-PART3-2-YIZHI
Sample Number: N/A
+TOF MS: 1.1485 to 1.1857 min from Sample 25 (XD-PART3-2-YIZHI) of 20211009 (recalibrated10).wiff different calibrations (DuoSpray ()), subtra...
Max. 4.7e6 cps
384.1592
386.1658
416.1491
Intensity, cps
m/z, Da
4r

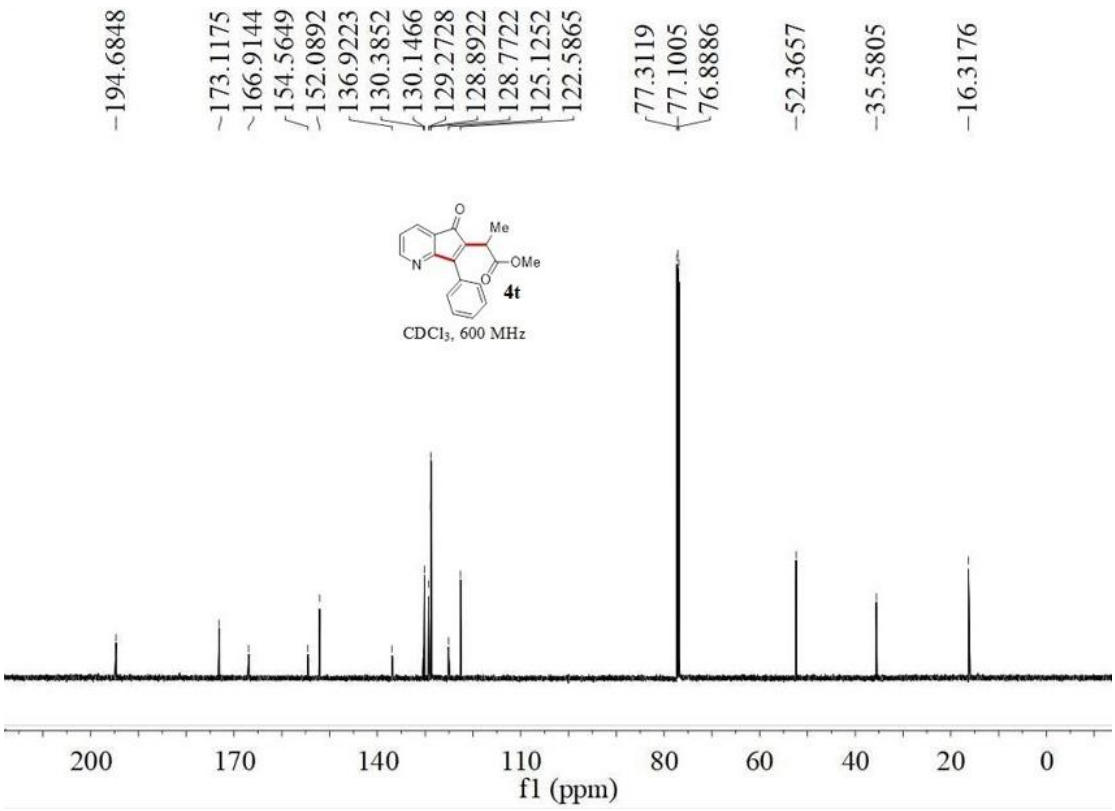

8.5080
8.4994
7.7355
7.7234
7.6201
7.6084
7.5542
7.5427
7.5300
7.5173
7.5053
7.2695
7.1719
7.1632
3.7196
3.7077
3.6958
3.6839
2.1513
1.4590
1.4470
Me
Me
4s
CDCl3, 400 MHz
1.00
1.10
2.05
2.11
1.10
1.00
1.00
3.06
3.07
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

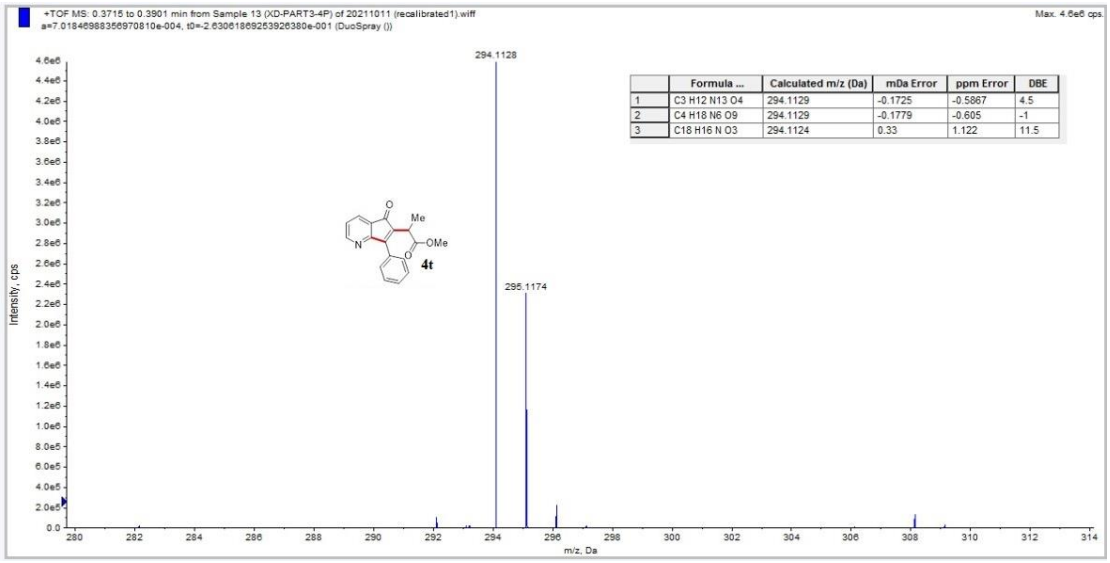

206.9360
195.1299
166.9089
155.6626
152.2458
137.2336
130.3181
129.3861
129.3182
128.8293
128.6732
122.6952
77.4271
77.1085
76.7902
44.1582
28.4379
15.4828
Me
Me
4s
CDCl3, 400 MHz
220
200
180
160
140
120
100
80
60
40
20
0
ppm

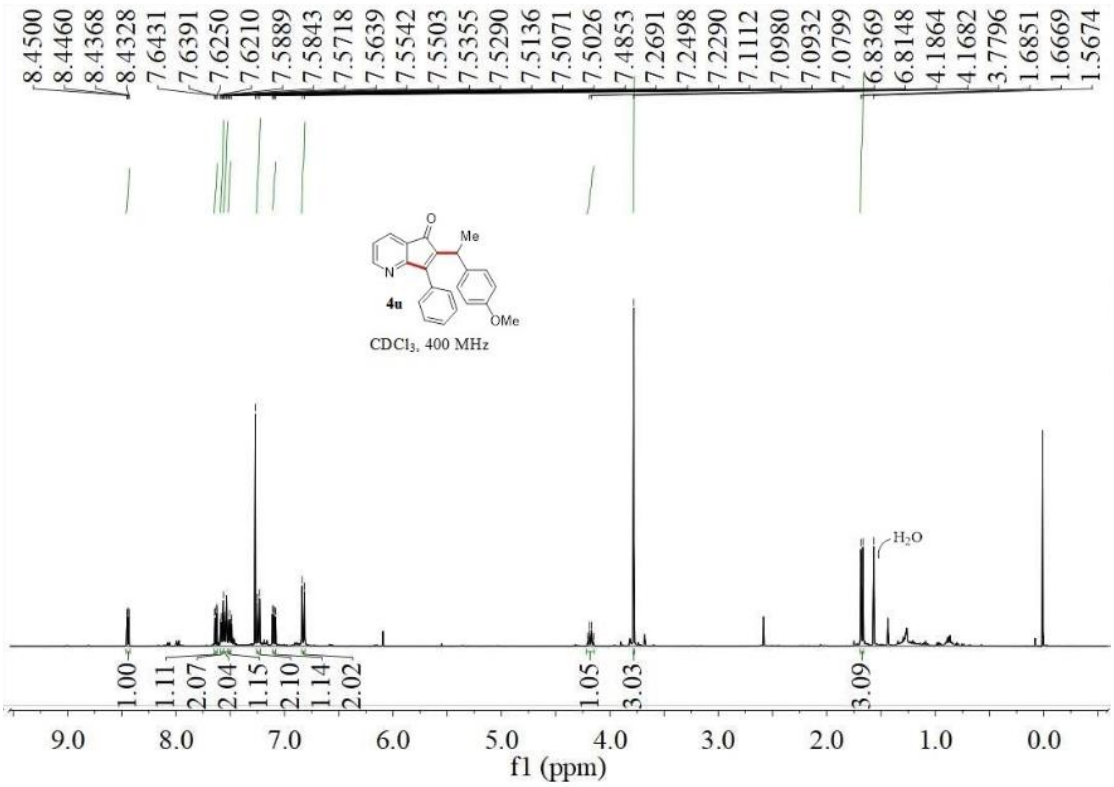


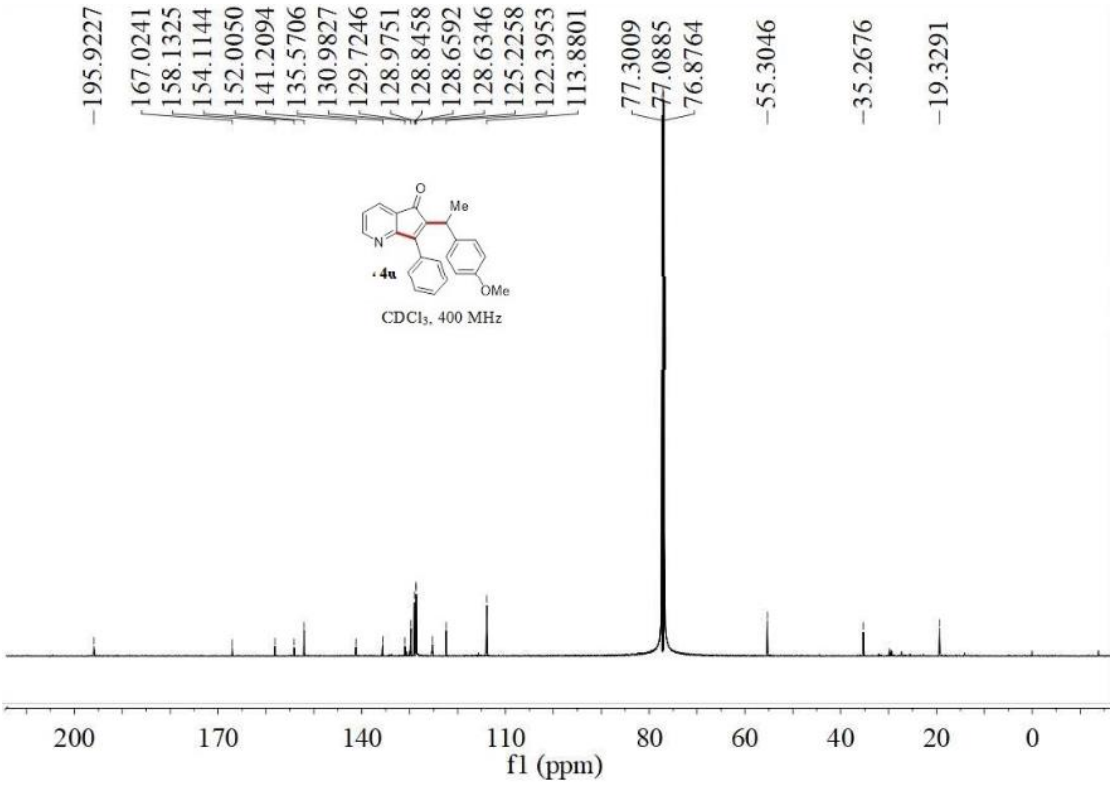
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C18 H16 N O2	278.1175	-0.1553	-0.5585	11.5
2	C10 H20 N3 O4 S	278.1169	0.4953	1.7812	2.5
3	C3 H12 N13 O3	278.118	-0.6579	-2.3656	4.5
4	C4 H18 N6 O8	278.118	-0.6633	-2.3849	-1
5	C3 H22 N2 O12	278.1167	0.674	2.4234	-6
6	C2 H16 N9 O7	278.1167	0.6793	2.4428	-0.5

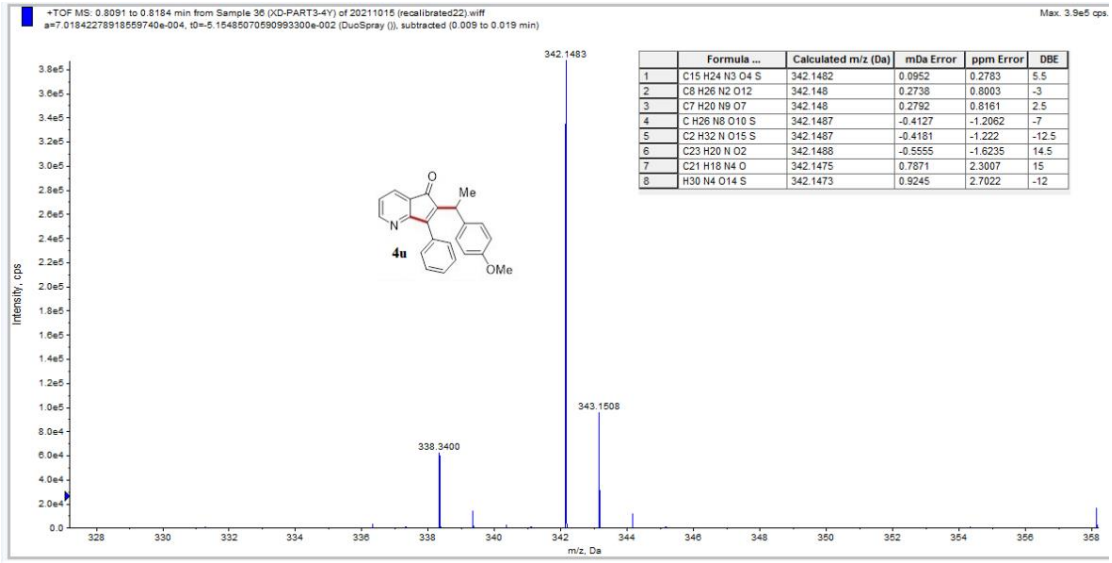



194.6848
173.1175
166.9144
154.5649
152.0892
136.9223
130.3852
130.1466
129.2728
128.8922
128.7722
125.1252
122.5865
77.3119
77.1005
76.8886
52.3657
35.5805
16.3176
Me
OMe
4t
CDCl3, 600 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)

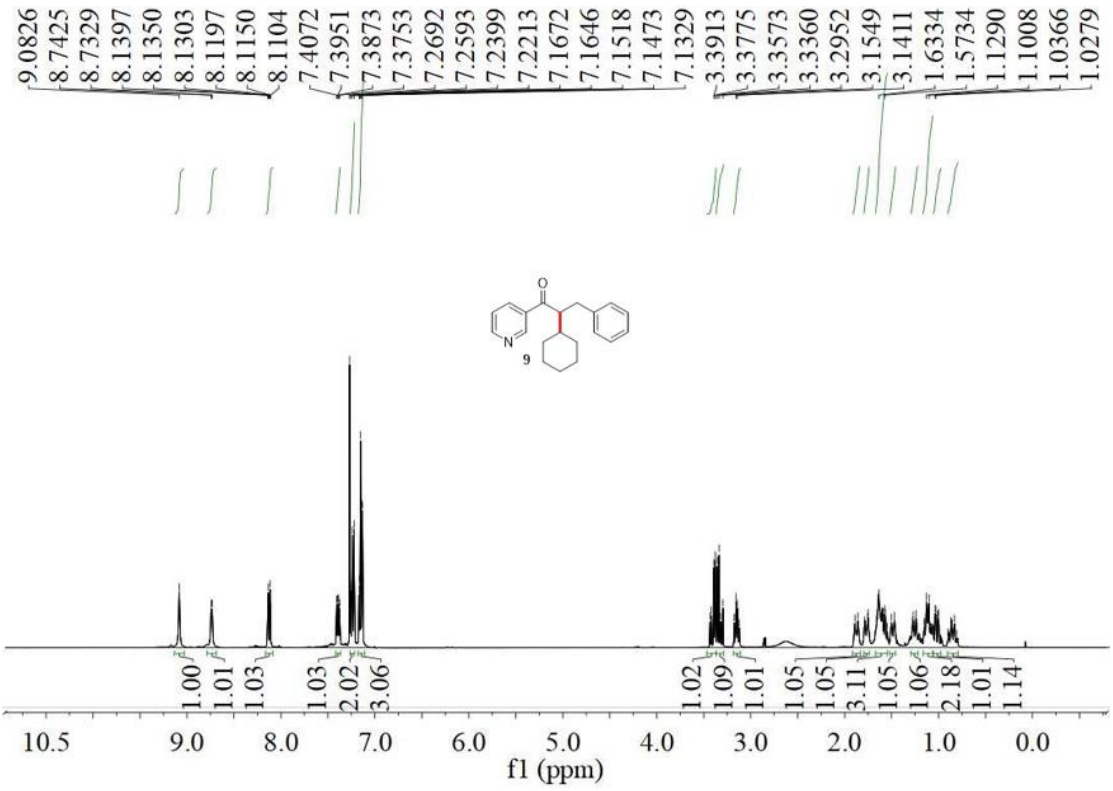

+TOF MS: 0.3715 to 0.3901 min from Sample 13 (XD-PART3-4P) of 20211011 (recalibrated1).wiff
Max. 4.6e6 cps.
294.1128
295.1174
Intensity, cps
m/z, Da
Me
OMe
4t
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C3 H12 N13 O4 | 294.1129 | -0.1725 | -0.5867 | 4.5
2 | C4 H18 N6 O9 | 294.1129 | -0.1779 | -0.605 | -1
3 | C18 H16 N O3 | 294.1124 | 0.33 | 1.122 | 11.5

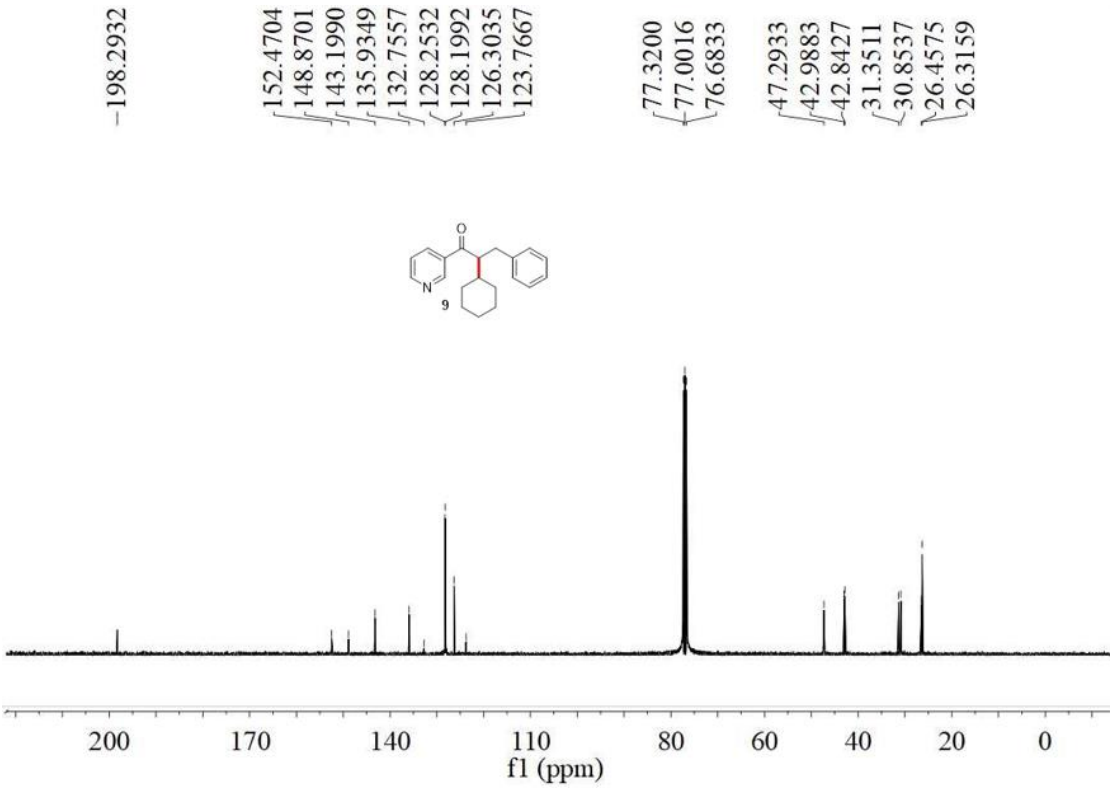

8.4500
8.4460
8.4368
8.4328
7.6431
7.6391
7.6250
7.6210
7.5889
7.5843
7.5718
7.5639
7.5542
7.5503
7.5355
7.5290
7.5136
7.5071
7.5026
7.4853
7.2691
7.2498
7.2290
7.1112
7.0980
7.0932
7.0799
6.8369
6.8148
4.1864
4.1682
3.7796
1.6851
1.6669
1.5674
Me
OMe
4u
CDCl3, 400 MHz
H2O
1.00
1.11
2.07
2.04
1.15
2.10
1.14
2.02
1.05
3.03
3.09
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

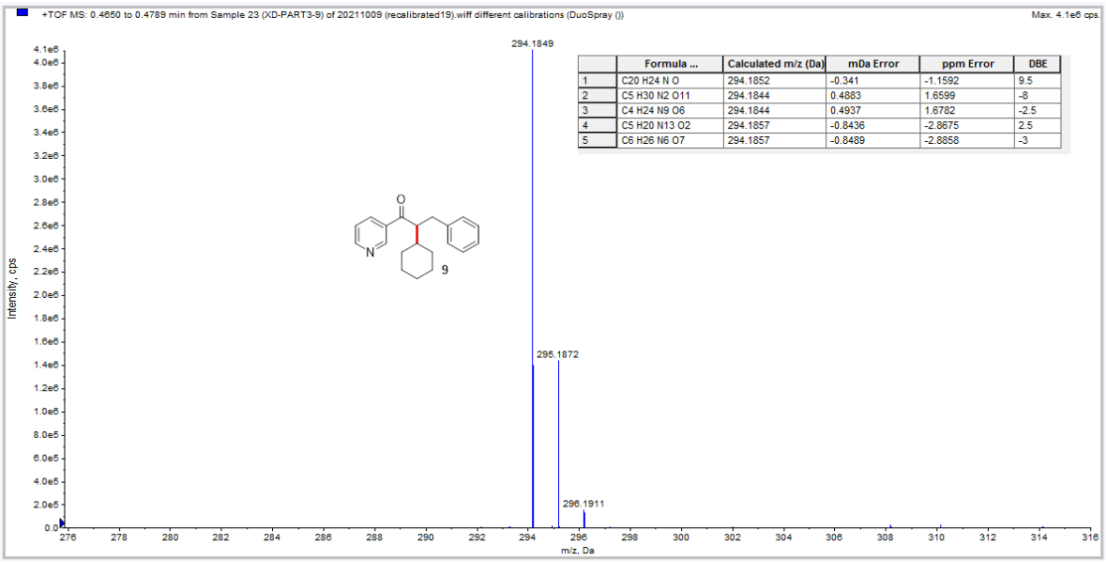

195.9227
167.0241
158.1325
154.1144
152.0050
141.2094
135.5706
130.9827
129.7246
128.9751
128.8458
128.6592
128.6346
125.2258
122.3953
113.8801
77.3009
77.0885
76.8764
55.3046
35.2676
19.3291
Me
OMe
4u
CDCl3, 400 MHz
200
170
140
110
80
60
40
20
0
f1 (ppm)



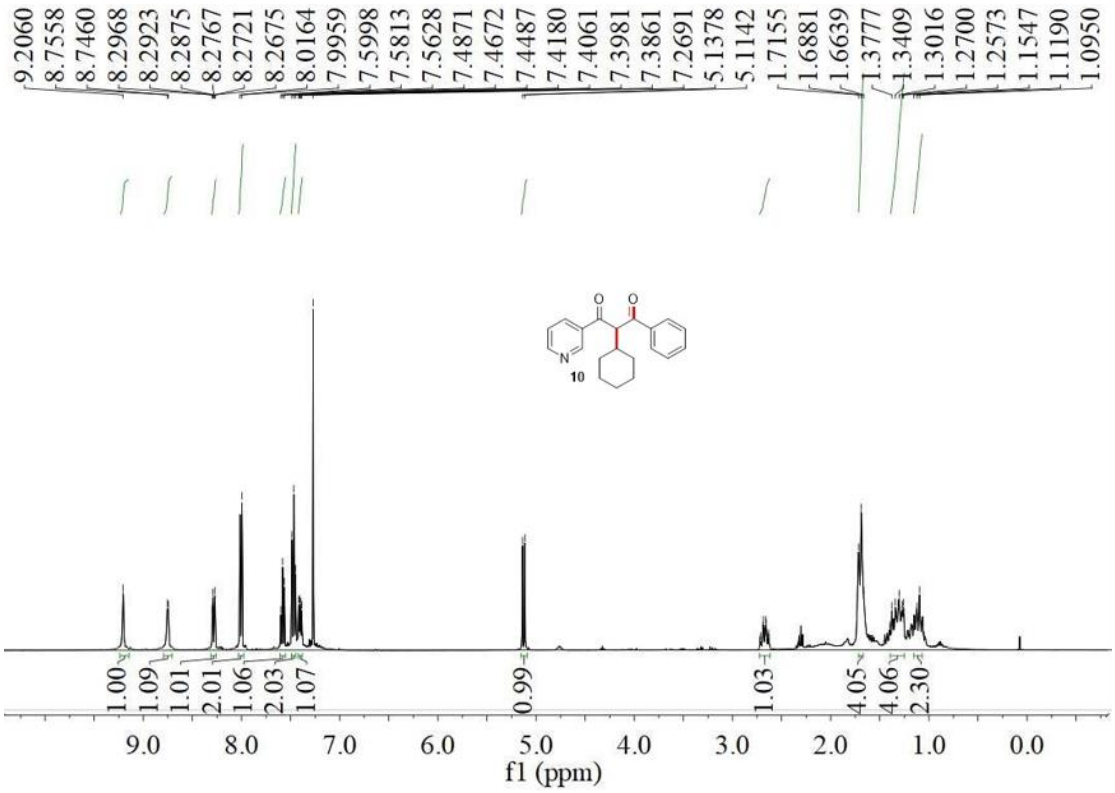
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C15 H24 N3 O4 S	342.1482	0.0952	0.2783	5.5
2	C8 H26 N2 O12	342.148	0.2738	0.8003	-3
3	C7 H20 N9 O7	342.148	0.2792	0.8161	2.5
4	C H26 N8 O10 S	342.1487	-0.4127	-1.2062	-7
5	C2 H32 N O15 S	342.1487	-0.4181	-1.222	-12.5
6	C23 H20 N O2	342.1488	-0.5555	-1.6235	14.5
7	C21 H18 N4 O	342.1475	0.7871	2.3007	15
8	H30 N4 O14 S	342.1473	0.9245	2.7022	-12

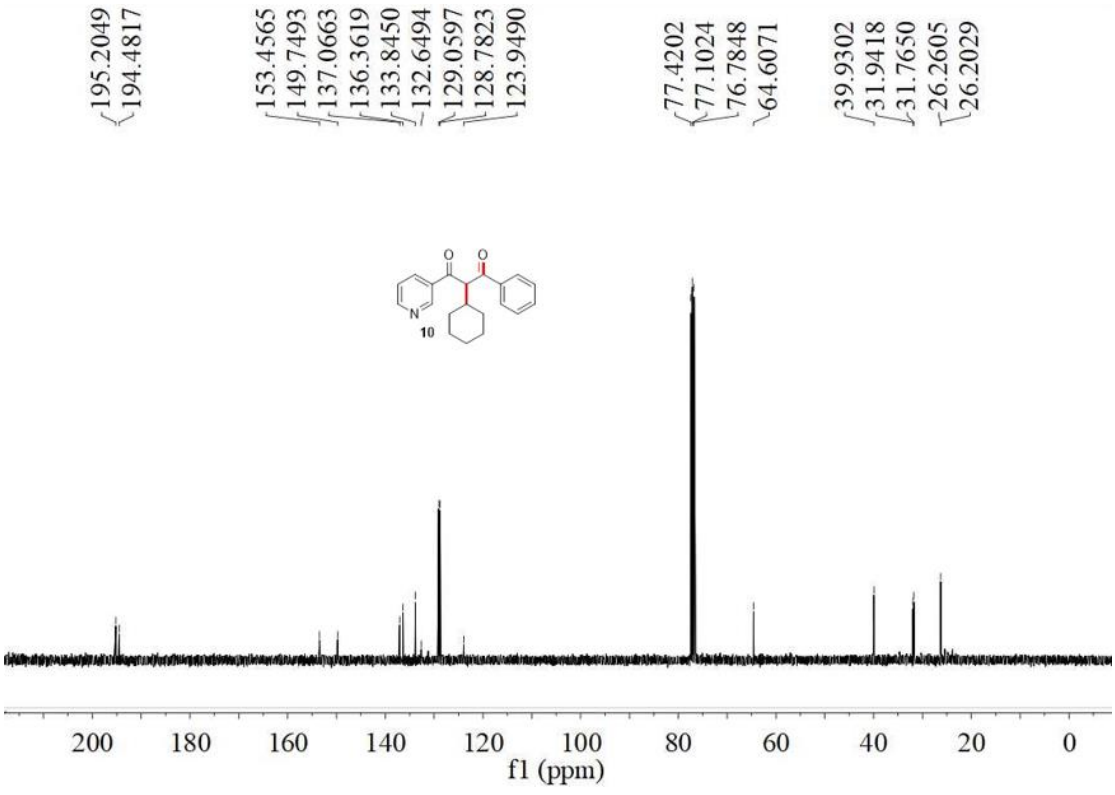


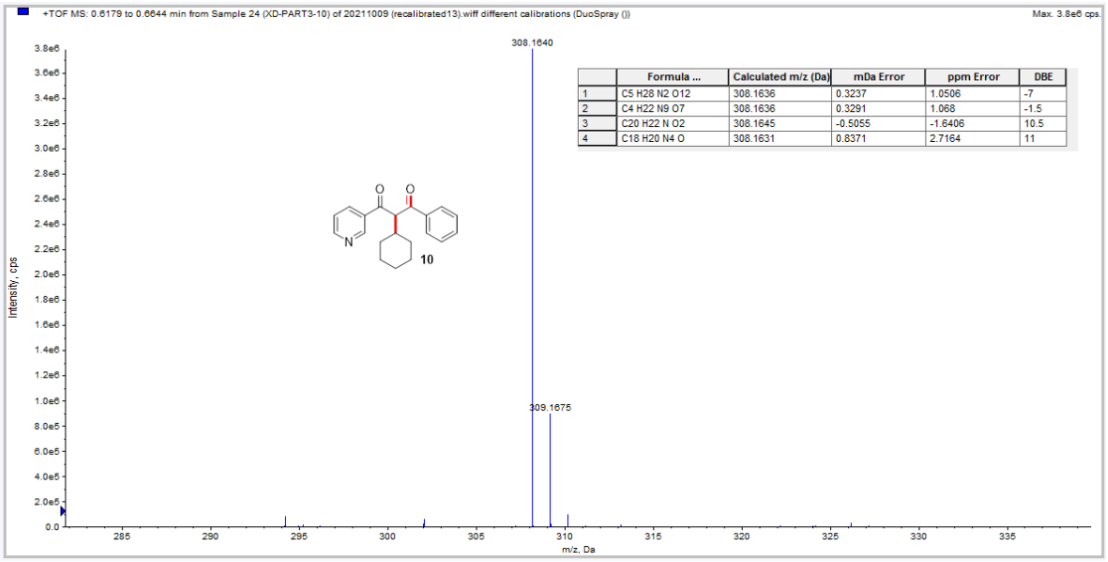




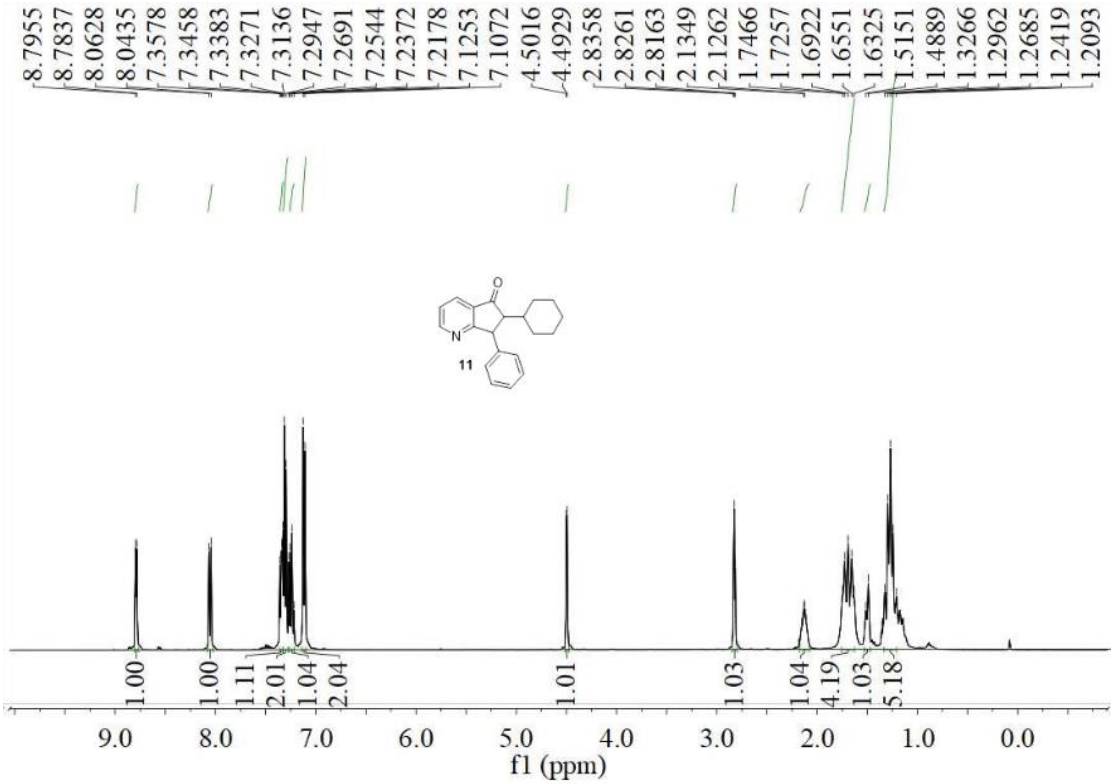
	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C20 H24 N O	294.1852	-0.341	-1.1592	9.5
2	C5 H30 N2 O11	294.1844	0.4883	1.6589	-8
3	C4 H24 N9 O6	294.1844	0.4937	1.6782	-2.5
4	C5 H20 N13 O2	294.1857	-0.8436	-2.8675	2.5
5	C6 H26 N6 O7	294.1857	-0.8489	-2.8858	-3

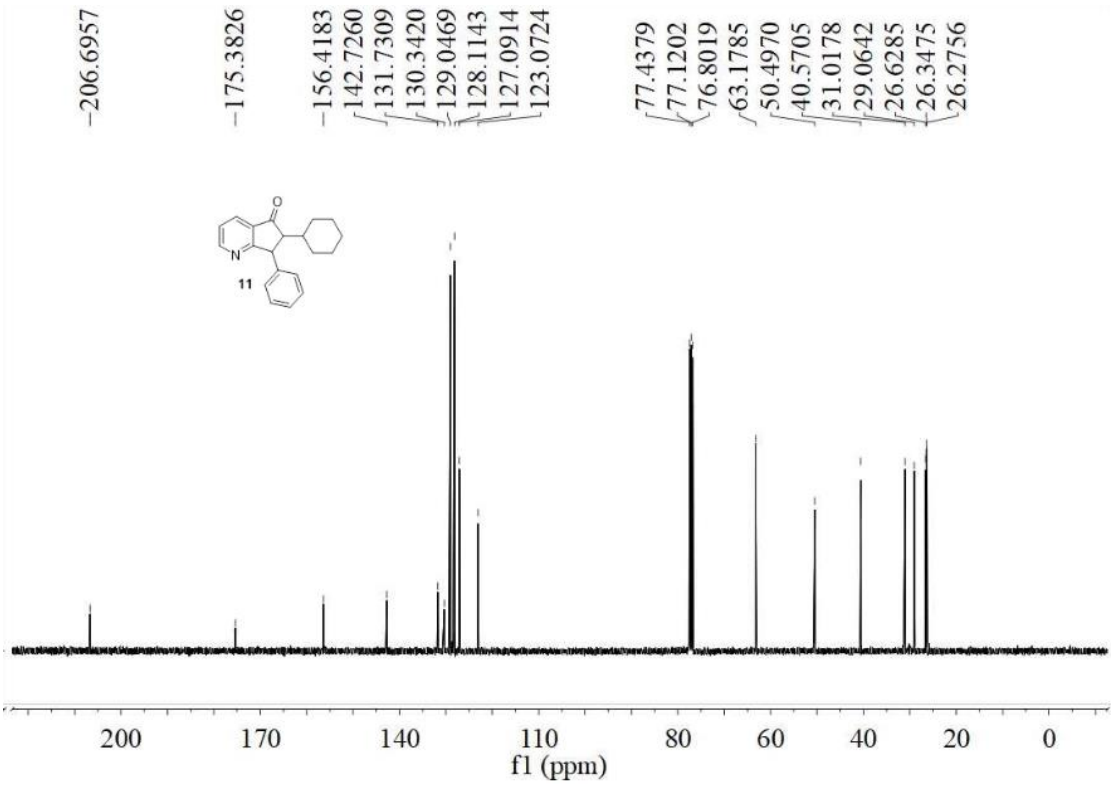

9.2060
8.7558
8.7460
8.2968
8.2923
8.2875
8.2767
8.2721
8.2675
8.0164
7.9959
7.5998
7.5813
7.5628
7.4871
7.4672
7.4487
7.4180
7.4061
7.3981
7.3861
7.2691
5.1378
5.1142
1.7155
1.6881
1.6639
1.3777
1.3409
1.3016
1.2700
1.2573
1.1547
1.1190
1.0950
10
1.00
1.09
1.01
2.01
1.06
2.03
1.07
0.99
1.03
4.05
4.06
2.30
9.0
8.0
7.0
6.0
5.0
4.0
3.0
2.0
1.0
0.0
f1 (ppm)

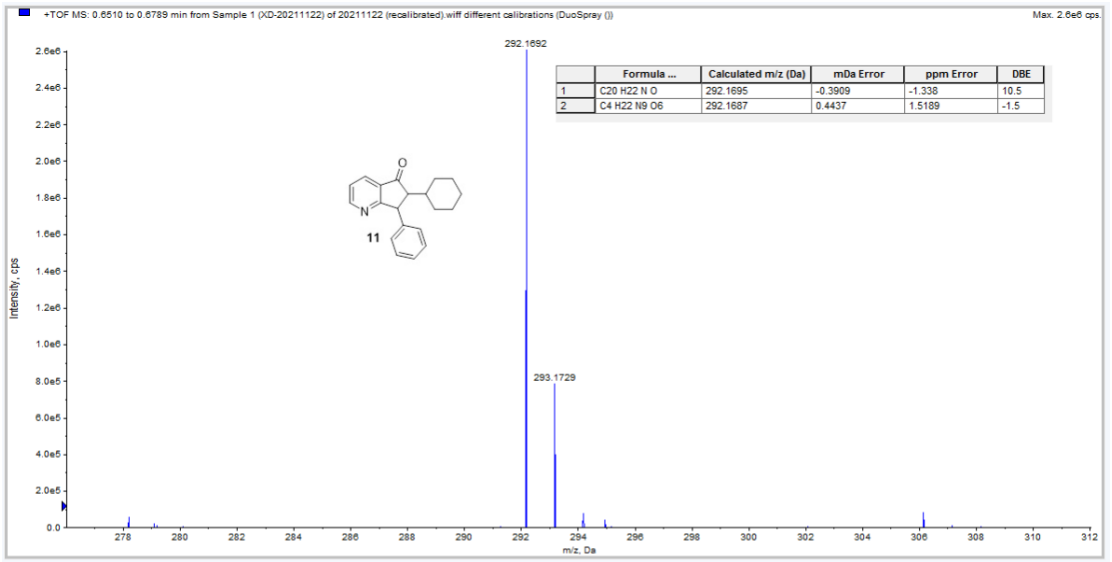

195.2049
194.4817
153.4565
149.7493
137.0663
136.3619
133.8450
132.6494
129.0597
128.7823
123.9490
77.4202
77.1024
76.7848
64.6071
39.9302
31.9418
31.7650
26.2605
26.2029
10
200
180
160
140
120
100
80
60
40
20
0
f1 (ppm)



	Formula ...	Calculated m/z (Da)	mDa Error	ppm Error	DBE
1	C5 H28 N2 O12	308.1636	0.3237	1.0506	-7
2	C4 H22 N9 O7	308.1636	0.3291	1.068	-1.5
3	C20 H22 N O2	308.1645	-0.5055	-1.6406	10.5
4	C18 H20 N4 O	308.1631	0.8371	2.7164	11




206.6957
175.3826
156.4183
142.7260
131.7309
130.3420
129.0469
128.1143
127.0914
123.0724
77.4379
77.1202
76.8019
63.1785
50.4970
40.5705
31.0178
29.0642
26.6285
26.3475
26.2756
11
200
170
140
110
80
60
40
20
0
f1 (ppm)


+TOF MS: 0.6510 to 0.6789 min from Sample 1 (XD-20211122) of 20211122 (recalibrated).wiff different calibrations (DuoSpray ())
Max. 2.6e6 cps.
292.1692
293.1729
Formula ... | Calculated m/z (Da) | mDa Error | ppm Error | DBE
1 | C20 H22 N O | 292.1695 | -0.3909 | -1.338 | 10.5
2 | C4 H22 N9 O6 | 292.1687 | 0.4437 | 1.5189 | -1.5
11
Intensity, cps
m/z, Da